EFFECTS OF PRESSURE DURING ROCK DEFORMATION

with particular reference to volume changes

by

James M. Edmond

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The work described in this thesis is entirely my own except where stated otherwise in the text.

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It is a particular pleasure to record my thanks to Dr. M.S. Paterson for his patient advice and constructive criticism throughout his supervision of this work.

Finally, thanks are due to my wife, Elizabeth, for her encouragement, and for typing this thesis.

James M. Edmond.

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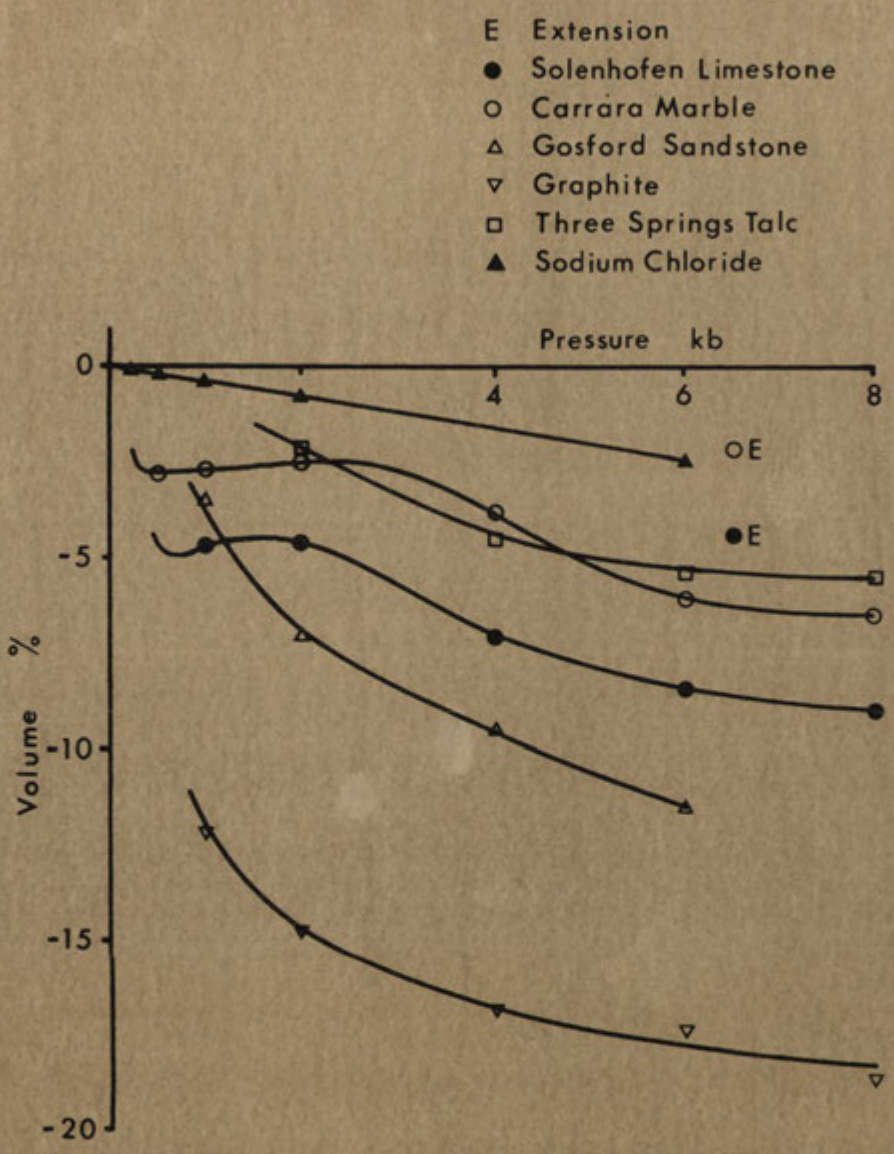

Frontispiece. Volume changes during reduction of confining pressure after deformation.

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CHAPTER I

INTRODUCTION

This thesis is concerned primarily with a study of the effects of confining pressure on the stress-strain properties, and of the mechanisms involved, when polycrystalline rock specimens are deformed beyond their elastic range.

A common method of determining stress-strain properties of a rock is by use of the so-called "triaxial test" in which a solid cylinder of the rock is subjected to a hydrostatic stress (the confining pressure) plus a superimposed axial stress (the differential stress) which may be either compressive or tensile according to whether the test is to be a compressive or an extensile one. The specimen is commonly sealed to prevent the pressure medium entering any pores or cracks. The apparatus may also have facilities to perform experiments at elevated temperatures and to introduce pore pressures into the specimen.

Most rocks in common experience are observed to be brittle, that is, any attempt to substantially change their shape results in gross fracturing and an associated stress drop. Increasing the confining pressure usually strengthens rocks and inhibits fracturing so that many rocks will flow and behave in a ductile fashion, that is they will undergo substantial permanent deformation which is uniformly distributed on the scale of the specimen. Most, if not all, deformations, regardless of the mechanism involved, will be found to be non-uniformly distributed if viewed on a small enough scale.

In the brittle range cataclastic mechanisms, namely sliding, rotation and fracturing, accommodate the deformation. In the ductile range deformation may proceed using cataclastic mechanisms or by intra-crystalline processes (crystal plasticity) such as slip and twinning and also by recrystallisation, or by various combinations of these. Paterson (1967) suggested that the gradient, tan ψ, (the "pressure sensitivity") of curves of the differential stress versus confining pressure at constant strain may be a convenient indicator of the mechanism of deformation. Tan ψ is closely related to the "coefficient of internal friction" of the material, the latter coefficient being more commonly used in failure criteria (Jaeger, 1962). Where cataclasis is the dominant mechanism tan ψ lies typically in the range 1 to 5, but when deformation proceeds by means of crystal plasticity this coefficient appears to be about two orders of magnitude lower. Intermediate coefficients would suggest a combination of cataclasis and crystal plasticity. Any discovery of crystal slip systems with a high pressure sensitivity would seriously upset this hypothesis. It should be emphasised that the pressure sensitivity of a slip system is not necessarily related to its intrinsic hardness. A soft slip system would therefore have its resistance to slip increased proportionally more than a hard slip system with the same pressure sensitivity.

The measurement of volume changes during deformation of a material may under certain circumstances provide useful

information about the mechanisms involved. In the absence of any porosity, if crystal plasticity is the sole mechanism, the deformation will proceed with no non-elastic volume changes, but if cataclasis is present, it will give rise to a non-elastic volume increase.

The transition from brittle to ductile behaviour with increasing confining pressure was first demonstrated in rocks by Adams and Nicolson (1901) and has been observed by numerous workers since using improved techniques, notably von Karman, (1911); Griggs, (1936); Robertson, (1955); Handin and Hager, (1957); Paterson, (1958); Heard, (1960); Mogi, (1966) and Byerlee, (1968). A higher confining pressure is required to give ductile behaviour during an extension test than during a compression test. Macroscopically the brittle-ductile transition seems to occur when the stress required to cause sliding on a shear fracture plane becomes greater than the stress required to initiate the fracture, the former stress being much more pressure sensitive than the latter. This in itself is not a sufficient explanation of the brittle-ductile transition for while it may explain the disappearance of the sudden stress drop at high confining pressures it does not explain the tendency for the deformation to become distributed throughout the specimen. This requires in addition, that there must be a strain hardening process,

leading to a stabilising effect, the nature of which is not fully understood in many cases. During plastic strain of a single crystal, defects cause dense entanglements of dislocations, and as the dislocation density increases a higher stress is needed to push new dislocations through the entanglement (McClintock and Argon, 1966). Polycrystalline materials are usually stronger and work harden more rapidly than single crystals because the grain boundaries provide very effective obstacles to dislocation movement and multiple slip is required if each crystal is to deform compatibly with its neighbours. For a crystal to undergo an arbitrary specified uniform deformation without volume change it must have five independent slip systems available (eg. von Mises, 1928; Taylor, 1938; Groves and Kelly, 1963; Paterson, 1969). In many cases this number of independent slip systems does not exist, or if they do, the prevailing conditions of temperature and pressure may prevent the activation of some of them and cataclastic mechanisms may become involved. Strain hardening when cataclastic mechanisms are involved may be due to comminution effects, (the smaller grains formed are stronger, and the differential load will be spread over an increasing number of points of contact), changes in packing, and the rate of volume change of the specimen.

Most triaxial testing of rocks at confining pressures over 1 kb has been concerned with stresses, axial strain and structural changes. Relatively little work has been done on the volume changes that occur during non-elastic deformation. Most of the early thought and work on volume changes was concerned with high porosity granular media, and volume changes are today considered of fundamental importance in soil mechanics. Unfortunately the theories evolved for granular media and the experimental techniques used cannot always be applied directly to the deformation of solid rock especially at high confining pressures. Simple models such as packed aggregates of hard spherical beads which may be useful when considering the behaviour of sand at low confining pressures are totally inadequate when considering for example a cemented sandstone deformed at a confining pressure of several kilobars. Use of such models (eg. Rowe, 1962; Orowan, 1966) neglects several mechanisms including those which give rise to strain hardening.

The earliest accurate measurements of volume changes resulting from deformation of solid rocks at atmospheric pressure were made by Bridgman (1949a), who observed volume increases before failure occurred in some low porosity rocks when compressed uniaxially. He used a method which permitted a direct measurement of the volume changes by immersing a

specimen in a liquid filled dilatometer provided with a capillary tube open to the atmosphere. Changes in volume of the contents of the dilatometer caused movement of the meniscus in the capillary. Such direct measurements of volume changes have several advantages over other methods. Assuming that the apparatus is properly calibrated, the accuracy of the results is independent of the porosity, permeability and local small scale inhomogeneities of deformation. The main disadvantage is that the method automatically integrates the volume changes throughout the entire specimen and gives a mean value. Its use is therefore limited if the deformation is localised. This, however, is a disadvantage in common with other volume change methods.

Handin, Hager, Friedman and Feather (1963) observed volume decreases during triaxial compression tests on rather porous limestone and sandstone, and observed volume increases in a lower porosity dolomite. Their method involved measuring variations in pore volume whilst maintaining a constant pore pressure. This, and similar methods, are limited in that they require good permeability and interconnection of all pores, conditions unlikely to be met by many rocks.

The most accurate results obtained so far came from Brace, Paulding and Scholz (1966) who used strain gauges bonded to the surface of the specimen. This method requires

considerable preparatory work on each specimen but gives accurate values for the surface strain within and a little beyond the elastic range of the specimens. They observed volume increases during triaxial compression tests on a granite, a marble and an aplite when differential stresses greater than about half the fracture stress were applied. Similar results were obtained by Matsushima (1960b, 1960c) using strain gauges, from uniaxial compression tests on a quartz monzonite and an olivine basalt, and from triaxial compression tests on a granite.

Other more qualitative methods have utilised various other phenomena. Obert and Duval (1942) were among the earliest workers in the rock mechanics field to study micro-seismic noise. More recently Scholz (1968) attempted to correlate micro-seismic events with the volume changes observed by Brace, Paulding and Scholz (1966). Other workers such as Tocher (1957) and Matsushima (1960a) have studied the variations in elastic wave velocities that occur during deformation.

Several workers such as Robertson (1955, 1960) and Paterson (1963) have measured the density of specimens at atmospheric pressure before and after triaxial tests. These measurements only give the net result of the volume changes which occur during increase of the confining pressure, deformation of the specimen, and reduction of the

confining pressure, and cannot distinguish between volume changes occurring during these three operations. Such measurements do provide an independent check, however, if the volume changes of the specimen can be monitored continuously throughout these three operations.

All the evidence suggests that volume changes during deformation of rocks may be a fairly general phenomenon and worthy of more attention than it has hitherto received. A large part of this thesis is concerned with the design of a dilatometer and its use in measuring volume changes during increase of the confining pressure, deformation of the specimen, and reduction of the confining pressure.

Note

The unit of pressure and stress in this thesis is the kilobar (1 kb = 10^9 dynes. cm^{-2} ≈ 14,500 lbf. $in.^{-2}$).

CHAPTER II

APPARATUS AND TECHNIQUES

ROOM TEMPERATURE PRESSURE APPARATUS

General arrangement

The apparatus, which is described in detail elsewhere (Paterson, 1964a), was designed by Dr. M.S. Paterson to deform materials under triaxial conditions at confining pressures up to 10 kb.

In brief, the apparatus consists of a pressure chamber C, which is attached to the baseplate B and contains the specimen assemblage, and a pair of externally yoked equal diameter pistons P entering at the ends (see Fig. II-1). This ensures that the volume and therefore the pressure in the chamber remains constant during any movement of the pistons. In addition, the forces on the pistons due to the pressure inside the chamber are cancelled out and the load cell L between the hydraulic jack J and the piston yoke therefore only measures the differential load on the specimen plus friction at the piston seals. All the non volume change experiments reported in this thesis are compressive, the lower piston being used to deform the specimen. The apparatus can also be used for non volume change extension experiments in which case the upper piston would be used to deform the specimen. The lower piston, however, was used for both compression and extension volume change experiments (see "Dilatometer" below). The term "extension experiment"

is used rather than "tension experiment" in this thesis since in all cases the net axial stress was compressive throughout the experiment although less than the confining pressure. No special mechanical gripping of the specimens was required, this being provided by the "suction" effect. The hydraulic jack J is controlled with a (coarse) hand pump, and a (fine) manual screw driven jack which controls the specimen strain rate. Another auxiliary jack (not shown) is used to move the piston yoke down against friction.

The pressure chamber is 1.27 in.diameter and 6.5 in. long (5.5 in. between the ends of the pistons) and is sealed at each end by plugs fitted with 'O'-ring seals. The pistons are also sealed inside the plugs with 'O'-ring seals (Paterson, 1962). Access to the pressure chamber is obtained by removing the plug E from the cross-head of the piston yoke and removing the upper plug and piston.

The pressure chamber is connected to an external pressure generating system consisting of a compressed air pump capable of priming the apparatus up to 2 kb, and a pressure intensifier. The pressure is increased above the priming pressure by a motor driven oil pump connected to the low pressure side of the intensifier. The pressure can be maintained at any desired level by setting the control contacts on the pressure recorder to that pressure, a manganin resistance gauge being used as the pressure transducer.

Kerosene was the usual pressure medium in my experiments, but petroleum ether was used as an alternative at the higher pressures when the viscosity of the kerosene became too great. This was the case when using the dilatometer (see later) with confining pressures greater than about 6 kb.

Piston displacement (10 mm full scale) and load (either 2,000 or 20,000 kg full scale) were recorded simultaneously on a two-pen recorder fed by a linear variable differential transformer (LVDT) D and the load cell L. Strain rate was standardised for all my experiments at approximately $4 \times 10^{-4} sec^{-1}$.

Specimen arrangement and jacketing

Fig. II.2 illustrates the specimen assemblage for non volume change compression experiments. The specimen S (10 mm diameter and 20 mm long) is sealed (to prevent the pressure medium entering any pores or cracks) between two steel end-pieces E with press fitted steel rings inside an annealed copper jacket with a wall thickness of 0.25 mm, or with rubber rings inside a red rubber jacket with a wall thickness of 1.6 mm. Rubber jackets sealed with rubber rings have the merits of simplicity and not requiring a correction for their load bearing capacity at low confining pressures. At pressures greater than about 5 kb, however, the rubber becomes glassy, its Young's Modulus increasing by about three

orders of magnitude (Paterson, 1964b; Weaver and Paterson, 1969 and substantial loads are borne by the rubber jackets. (See Appendix). The sealed specimen is deformed by the lower piston P between the lower anvil F and the upper reaction piece R, the tubular container A facilitating insertion and withdrawal of the assemblage.

Calibration

The LVDT displacement transducer was calibrated using a Wild cathetometer with a reading accuracy of ± 0.02 mm. Some slight non-linearity was discovered in the LVDT output but this only gave rise to a maximum error of 1% of any displacement measured.

The Philips load cells were checked against an Amsler box having an accuracy of ± 0.5%.

The manganin coil pressure transducer with a dummy gauge for temperature calibration was checked against a 3 kb Bourdon gauge having an accuracy of ± 0.5%. The manganin gauge was then assumed to be linear up to 10 kb after Bridgman (1949b). A calibration error up to ± 2% may therefore be expected at 10 kb.

Derivation of stress strain curves

Corrections from calibration experiments were applied a) for the effects of friction at the piston seals (determined at pressure before and after each experiment, this was always less than 2% of the total load on the two pistons),

b) the load borne by the specimen jacket, (see Appendix), and c) elastic distortion of the apparatus (0.11 mm per 1000 kg of differential load in excess of piston friction).

The specimen strain was calculated by dividing the change of length ΔL due to the differential load by the original undeformed length of the specimen at atmospheric pressure, L_o.

i.e. Expressed as a percentage, strain = $\frac{\Delta L}{L_o} \times 100\%$.

Compressive strains are positive.

The differential stress was calculated by dividing the differential load by the cross-sectional area of the specimen at that strain assuming uniformly distributed deformation and no volume change.

i.e. Differential stress = $\frac{\text{Differential load}}{A_o}$ (1 - strain)

where A_o is the original cross-sectional area of the undeformed specimen at atmospheric pressure. Compressive stresses are positive.

Accuracy

The assumption of uniform deformation and no volume change in calculating the actual cross-sectional area at any stage during deformation clearly involves errors which are characteristic of that particular experiment. This approximation for the true differential stress gives a better indication of whether the specimen fabric is itself strain hardening or not. Errors in the stress-strain (as defined)

curves are not expected to be greater than about 3% of the differential stress except during about the first 2% of strain where some uncertainty exists. The confining pressure could be held constant to within ± 2% during an experiment. In general, these errors are small compared with the irreproducibility found to occur between similar rock specimens tested under identical conditions.

HIGH TEMPERATURE PRESSURE APPARATUS

General arrangement

The apparatus was designed by Dr. M.S. Paterson to deform materials under triaxial conditions at confining pressures up to 10 kb and temperatures up to 1000°C. It is described in detail elsewhere (Paterson, 1970). Compressive experiments only, were performed at elevated temperatures.

The apparatus (see Figs. II-3,4) is basically similar to the room temperature apparatus described above, the main points of difference being the presence of an internal furnace, an internal load cell and the use of an argon pressure medium. Priming pressure up to 2 kb is obtained with a series of pressure exchangers and is then increased and maintained at any desired level by pumping on a pressure intensifier after setting the control contacts on the pressure recorder to that

level. A manganin coil pressure transducer with a dummy coil for temperature compensation is used.

The temperature is controlled manually using a Variac transformer supplied with a stabilised voltage, and is measured with a series of thermocouples. The use of an internal load cell obviates the need for piston friction corrections. The strain rate is controlled by an electrically powered variable speed screw-driven jack and was standardised at approximately $4 \times 10^{-4} sec^{-1}$ for all my experiments. Displacements are measured with an externally mounted LVDT.

Differential load and displacement were simultaneously recorded on one two-pen recorder, and pressure and temperature on another.

Specimen arrangement and jacketing

Specimens 10 mm in diameter and 20 mm long were sealed inside 0.25 mm wall thickness annealed copper jackets between the upper and lower pistons with press fitted steel rings. Either a solid or hollow upper piston could be used, the latter venting the specimen to the atmosphere and preventing any build up of pore pressure in the specimens. To prevent extrusion of weak specimens up the 1/16 in. diameter hole in the hollow piston, a finely perforated molybdenum alloy disc was interposed between the specimen and the piston.

Calibration

Similar to the room temperature apparatus. The temperature calibration was checked regularly using a jacketed hollow steel specimen and hollow upper piston into which a thermocouple was inserted.

Derivation of stress strain curves

Similar to the room temperature apparatus except that no correction for piston friction was required. For loads borne by copper jackets at various temperatures see Appendix.

Accuracy

Similar to the room temperature apparatus. An error of ± 3% of the nominal temperature may be expected in these experiments.

DILATOMETER

Principles of operation

This apparatus was designed to measure volume changes occurring during deformations of up to 25% in compression at confining pressures of up to 10 kb at room temperature. Similar experiments in extension were performed using smaller specimens and the "push-pull converter" described later. The dilatometer, (Figs. II-5, 6, 7) fits inside the room temperature pressure apparatus described above.

For compression experiments the specimen S, 10 mm diameter and 20 mm long, is sealed with press fitted rings between two steel endpieces E inside an annealed copper jacket J of 0.25 mm wall thickness. This specimen assembly is enclosed within a brass bellows arrangement A which is filled with water (plus sodium dichromate to prevent corrosion) for experiments at confining pressures up to and including 8 kb. (In the event of the apparatus being used at higher confining pressures an alternative fluid would have to be used as water solidifies at about 9 kb at room temperature.) The specimen is compressed by the piston P between the lower anvil F and the upper reaction anvil R, which is made from ASSAB 8407, a 5% Cr-Mo-V steel, A.I.S.I type H 13, hardened and tempered to R_c.50. In the original design this upper anvil was constructed of a harder and more brittle steel and some failures were experienced due to the upper endpiece punching through the anvil. Any volume change of the specimen therefore causes an expulsion or intake of water through the holes H, which connect bellows A to bellows B, both bellows being full of water. The displacement of the free upper end of bellows B is related to the volume change of the specimen. As the specimen is shortened so is bellows A, and this causes an expulsion of water into bellows B. Bellows A and B have the same cross-sectional area so the difference in movements

between the lower end of bellows A and the upper end of bellows B is directly proportional to the volume change of the specimen. By means of a suitable yoke X , the core of a LVDT, T, (040 MS-L type supplied unpotted together with a CAS-2500 type carrier amplifier demodulator system by Schaevitz Ltd., America) can be attached to the lower end of bellows A and the barrel of the LVDT can be attached to the upper end of bellows B. The signal from the LVDT passes out of the pressure vessel through the top plug of the apparatus which has insulated and pressure sealed leads through it. Relative movement between the ends of the bellows can thus be measured with a high degree of accuracy.

The water in the bellows is itself compressible (specific volume at 8 kb and 20°C = 0.826 $cm.^3\ g^{-1}$) so when deliberate changes of the confining pressure are made, or small fluctuations occur, the new pressure is immediately transmitted to the water via a small movement of the upper free end of bellows B. The volume of water in bellows A and B would therefore change with the confining pressure and cause displacements to be measured at the LVDT. To prevent this happening, a pressure variation compensation system is incorporated. This consists of another bellows C and yoke Y which cause the core of the LVDT to move by exactly the same amount and in the same direction as the LVDT barrel in

response to any pressure fluctuation. During the design stage careful matching of the water volumes with the bellows' diameters is essential but small adjustments to the volumes can be made. This system also permits the measurement of specimen volume changes as the confining pressure is raised and lowered before and after the deformation.

Push-pull converter

This was designed and constructed to allow extension volume change experiments to be performed using the same dilatometer. The specimen, 7 mm diameter and 13 mm long, is solder sealed (there was insufficient space available to use steel sealing rings) inside an annealed copper jacket of 0.25 mm wall thickness between two steel endpieces. When the converter is assembled a compressive load applied at the ends is converted to an equal extensile load at the specimen, (Fig. II.8). This converter fits inside bellows A of the dilatometer in place of the usual specimen assemblage.

Calibrations

a) Dilatometer LVDT. This was done with the dilatometer LVDT, T, in position at various confining pressures. No specimen was used and a perforated screw cap was used in place of the normal screw cap C and the lower anvil F; bellows A and B were filled with the pressure medium (kerosene, or petroleum ether for 8 kb confining pressure). By slowly

pushing up on the perforated screw cap with the piston P, the core of the dilatometer LVDT was made to move out of the barrel of the LVDT due to the yoke X by the same amount as the piston displacement which was measured with the pressure apparatus LVDT (D in Fig. II.1), there being no movement of bellows B. The change in voltage output of the dilatometer LVDT per unit length of core displacement was thus determined. Knowing the effective cross-sectional area of bellows B, the output of the LVDT per unit volume change for a standard size specimen was calculated, and the d.c. voltmeter calibrated in percentages of actual volume change with respect to the original bulk volume of the specimen at atmospheric pressure (volume decrease being defined as a positive volume change).

With a 10 mm diameter and 20 mm long specimen the output was 0.087 volt per 1% volume change of the specimen. With a 7 mm diameter and 13 mm long extension specimen the output was 0.028 volt per 1% volume change of the specimen. (These calibrations were found to be unaffected by confining pressure). A range of more than $\pm$ 25% volume change was available before non-linearity of the LVDT characteristics became significant. The resolution of the d.c. voltmeter was 0.01 volts, or 0.005 volts with extra care. The theoretical resolution of the dilatometer was about 0.1% volume change for compression and about 0.2% volume change for extension experiments.

b) Change of confining pressure (without differential stress). Using a tungsten carbide specimen, (a small correction was made for the volume change of the specimen), the pressure was cycled and the volume of water in the pressure compensating system optimised. Some difficulty was experienced in the region 0-0.2 kb due to the presence of small air bubbles and the collapse of the copper jacket into surface indentations on the specimen.

c) Differential stress (without deformation or volume change). Also determined using a tungsten carbide specimen and applying a differential load of 10,000 kg, equivalent to about 12.5 kb on a 10 mm diameter specimen (small corrections were made for the deformation and volume change of the specimen). This gave a value of 0.13 mm per 1000 kg differential load in excess of friction for the elastic distortion of the apparatus and confirmed that no anomalous volume changes would be observed resulting solely from the application of a differential load.

d) Deformation (without volume change). Determined by deforming annealed copper specimens at various confining pressures (small corrections were made for volume changes of the specimens). Anomalous volume changes were recorded during these experiments but they were reproducible at all confining pressures and a correction of -0.06% volume change

per 1% deformation was required. This is caused by slight variations in the effective diameters of bellows A and B as they are respectively compressed and extended during deformation. Heat produced during deformation was quickly conducted away and did not cause any difficulties.

Accuracy

a) During deformation and removal of differential load before reducing confining pressure. Errors of not more than 0.15% volume change for compression and 0.30% volume change for extension experiments may be expected after 20% deformation. These were small compared with the irreproducibility found to occur between similar rock specimens tested under identical conditions.

b) During increase and decrease of confining pressure. Due to the presence of air bubbles, imperfectly fitting copper jackets and small variations of volume between specimens (giving rise to small variations in the volume of water in bellows A and B), larger errors occur in these measurements. The experiments with annealed copper specimens showed that errors of up to 0.4% volume change after compression experiments and up to 1% volume change after extension experiments could be expected, these being independent of the actual confining pressure at which the experiment was performed.

Density measurements

Bulk densities were determined for specimens before and after deformation by weighing the specimens successively in air and water. The specimens were sealed with paraffin wax of known density (0.910 $g.cm^{-3}$) to prevent water entering the pores. The sensitivity of this method was about 1 part in 1000 which was adequate, the actual variations between individual specimens being greater than this.

The porosity of undeformed specimens was calculated using assumed values for the solid's density and the relation:-

$$\text{Porosity} = \frac{\text{Volume of voids}}{\text{Total volume}} \times 100\%$$

$$= \frac{\text{Solids density - Bulk density}}{\text{Solids density}} \times 100\%$$

The actual volume change of specimens as a result of an experiment (the increase and decrease of confining pressure as well as the actual deformation) was calculated using the relation:-

$$\text{Volume change} = \frac{\text{Original volume - Final volume}}{\text{Original volume}} \times 100\%$$

$$= \frac{\text{Final bulk density-Original bulk density}}{\text{Final bulk density}} \times 100\%$$

i.e. dilation is negative according to my convention.

X-RAY EQUIPMENT

Textural information was obtained with a Philips texture goniometer and using a technique based on that developed by Schulz (1949a; 1949b).

A Philips diffractometer was used to identify minerals in powdered specimens.

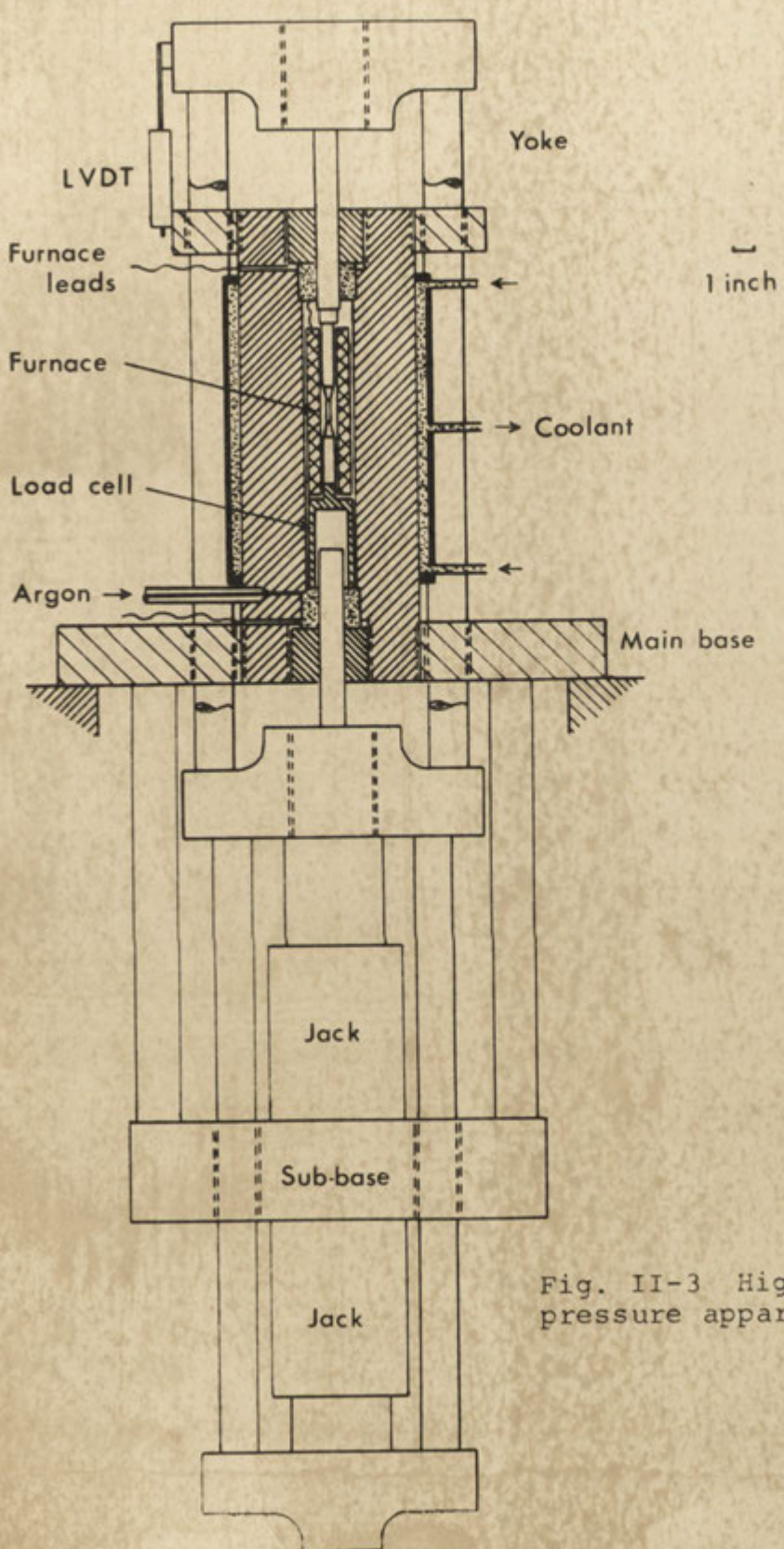


Fig. II-3 High temperature pressure apparatus.

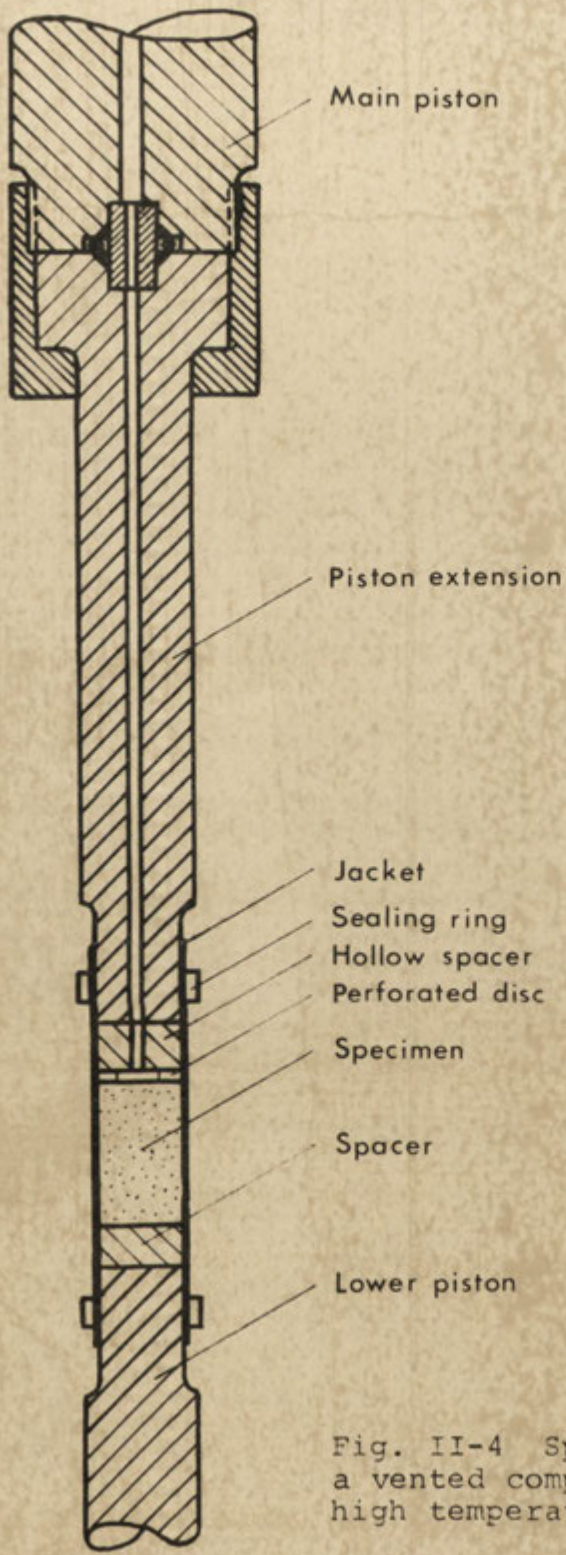


Fig. II-4 Specimen assemblage for a vented compression experiment at high temperature.

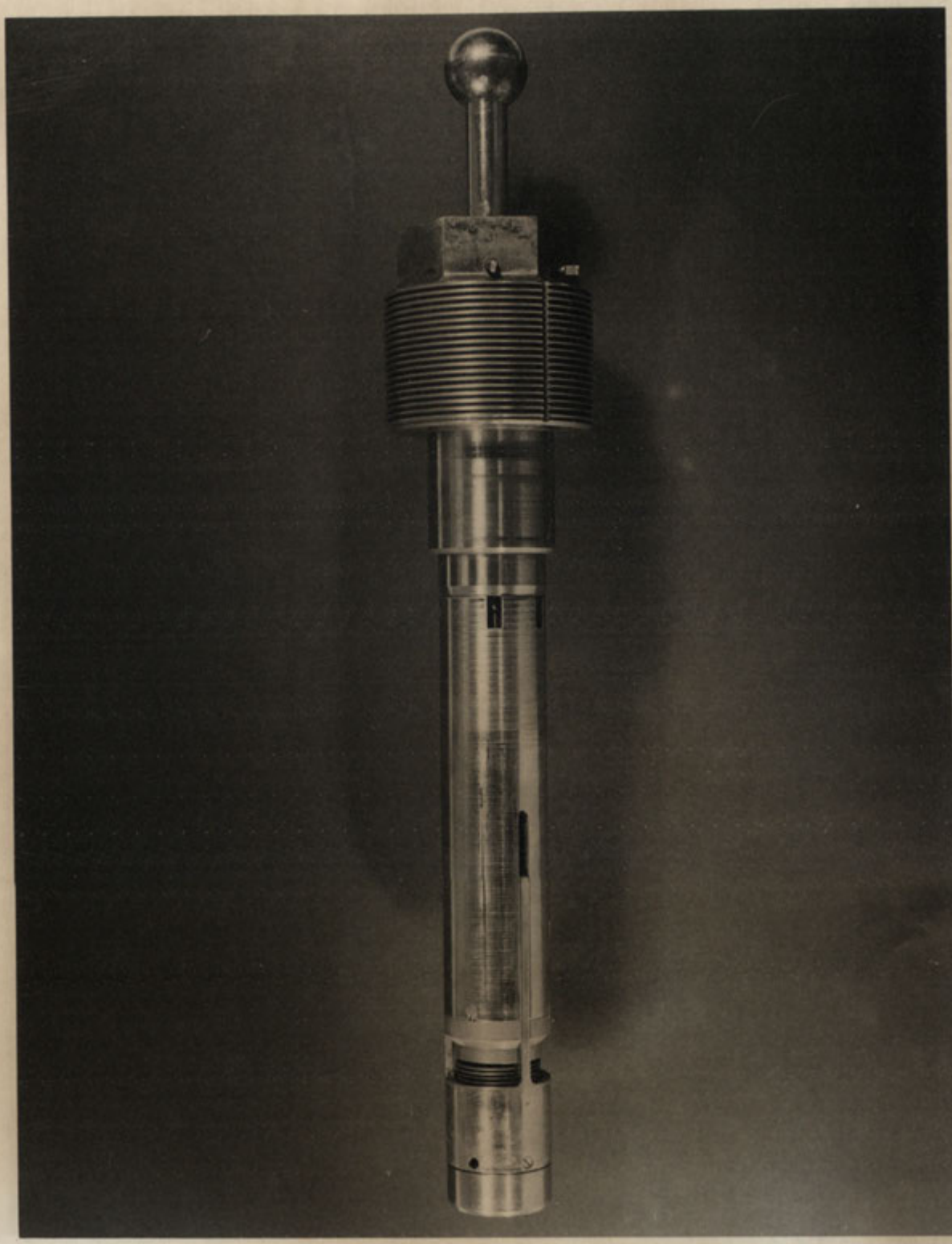
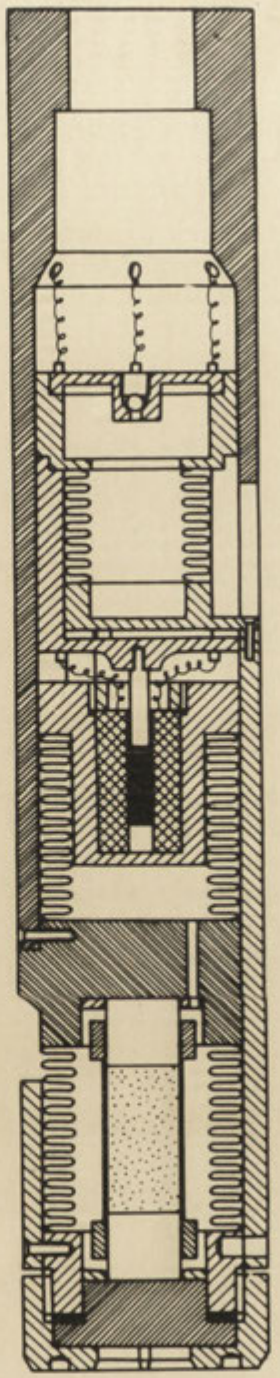


Fig. II-5 Dilatometer



1 inch

Fig. II-6 Dilatometer in Section

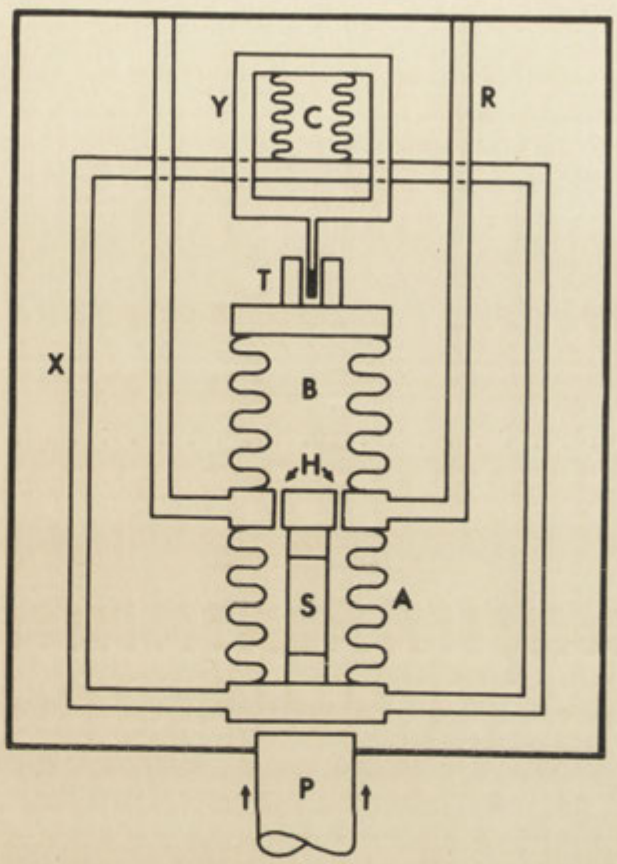
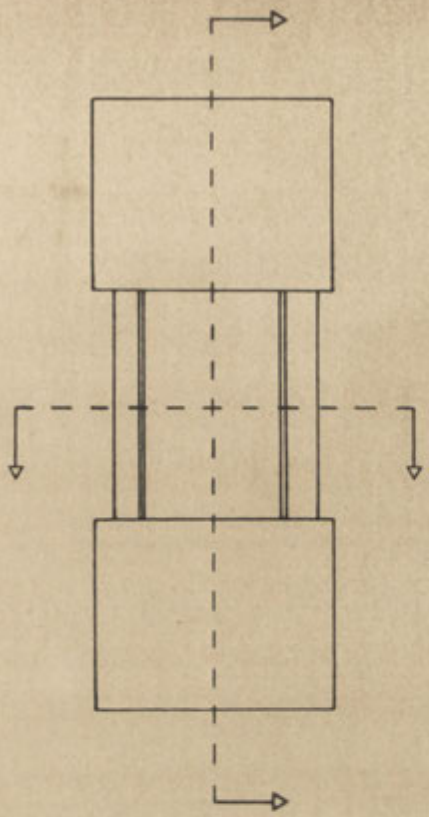
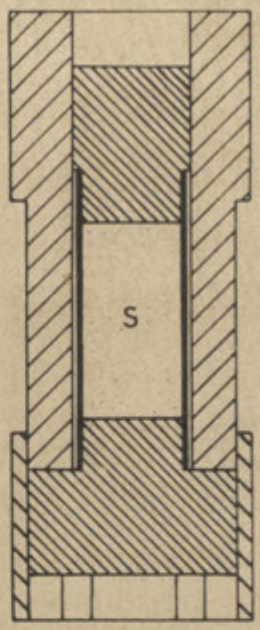
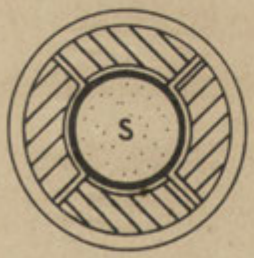


Fig. II-7 Schematic Drawing of Dilatometer







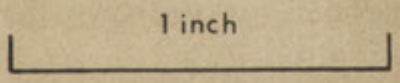


Fig. II-8 Push-pull converter

CHAPTER III

DEFORMATION
OF
A LIMESTONE, A MARBLE AND A SANDSTONE

Introduction

Solenhöfen limestone and Carrara marble are undoubtedly two of the "classic" materials in the field of rock deformation. It seemed logical therefore that these rocks, whose stress-strain properties were already relatively well documented, should be used during any attempt to introduce a new dimension into this field. Gosford sandstone was also selected as being representative of another important class of rocks which are known to deform in a ductile manner at confining pressures well within the range of our apparatus.

Materials

Solenhöfen limestone (Bavaria) A very pure fine grained (about 0.01 mm) limestone. The bulk density was 2.56 g. cm^{-3} giving a theoretical porosity of about 5.9%. No anisotropy was detected on the stress-strain curves during preliminary experiments in which three specimens cored in orthogonal directions were deformed at a confining pressure of 4 kb. Specimens were made for extension and compression experiments by grinding 7 mm and 10 mm diameter diamond drill cores to lengths of 13 mm and 20 mm respectively. Specimens were air dried at room temperature for several weeks before use.

Carrara marble (Italy) A very pure true marble with a grain size of about 0.1 mm. The bulk density was

2.69 g. cm^{-3} giving a theoretical porosity of about 1.1%. No anisotropy was detected on the stress-strain curves during preliminary experiments in which three specimens cored in orthogonal directions were deformed at a confining pressure of 4 kb. Specimens for extension and compression experiments were made by grinding 7 mm and 10 mm diameter diamond drill cores to lengths of 13 mm and 20 mm respectively. Specimens were air dried at room temperature for several weeks before use.

Gosford sandstone (N.S.W., Australia) Consisted of weakly cemented quartz and feldspar crystals about 0.2 mm in size in a matrix of mica and clay minerals. The bulk density was 2.45 g. cm^{-3} giving a theoretical porosity of about 13%. This sandstone is anisotropic and very variable from place to place. All specimens used for these experiments were cored from the same block in the same direction using a 10 mm diameter core diamond drill and ground to lengths of 20 mm. Specimens were air dried at room temperature for several weeks before use.

Techniques

Solenhöfen limestone Room temperature volume change compression and extension experiments as described in Chapter II. Three specimens were deformed in compression at each of the confining pressures 1 kb, 2 kb, 4 kb and

8 kb, and three in extension at a confining pressure of 6.5 kb. A further three specimens were deformed at a confining pressure of 10 kb in room temperature non volume change experiments using annealed copper jackets with a wall thickness of 0.25 mm.

Carrara marble Room temperature volume change compression and extension experiments as described in Chapter II. Three specimens were deformed in compression at each of the confining pressures 0.5 kb, 1 kb, 2 kb, 4 kb, 6 kb and 8 kb, and three in extension at a confining pressure of 6.5 kb. A further three specimens were deformed at a confining pressure of 10 kb in room temperature non volume change experiments as for the Solenhöfen limestone above.

Gosford sandstone Room temperature volume change compression experiments as described in Chapter II. Three specimens were deformed at each of the confining pressures 1 kb, 2 kb, 4 kb and 6 kb.

Results

Solenhöfen limestone The transition from brittle to ductile behaviour occurred in compression at a confining pressure between 1 kb and 2 kb and corresponded with the change from a falling to a continually rising stress-strain curve. The means of the stress-strain curves are drawn in Fig. III-1; the vertical marker indicates the scatter which was

about the same at all confining pressures. In the confining pressure range 2 kb to 10 kb and for compressive strains up to 4%, increasing the confining pressure apparently reduced the flow stress of the rock. This effect has been commented upon previously (Paterson, 1964a) and demonstrates that increasing the confining pressure does not invariably strengthen a rock. The increasing confining pressure apparently makes some of the pore volume more prone to elimination when the differential stress is applied. At strains in excess of about 4% the rate of strain hardening and the differential stress increase with increasing confining pressure but at a reducing rate. These effects can be illustrated by plotting the differential stress, at various strains, against the confining pressure (Fig. III-2).

The extension experiments done at a confining pressure of 6.5 kb had to be terminated at about 13% strain because the specimens began to "neck" and failure was imminent. (Necking was indicated on the load-displacement record by a decrease in the differential load on the specimen). The mean stress-strain curve for the extension experiments is also drawn on Fig. III-1 although according to our convention the differential stresses and strains in this case should be negative. The scatter of the original stress-strain curves for the extension experiments was similar to that for

the compression experiments. The extension curves were significantly higher at small strains but the strain hardening rate at large strains was lower than for compression curves at similar confining pressures. This effect, which has not yet been satisfactorily explained, was also observed by Paterson (1964a).

During compression experiments with confining pressures between 1 kb and 8 kb the specimens initially decreased in volume. This was followed by large volume increases at a decreasing rate at the lower end of this pressure range and further volume decreases also at a decreasing rate at the higher pressures (Fig. III-3). The scatter of the original curves is indicated by the vertical marker. Deformation at constant volume would be expected at a confining pressure about 2.5 kb. Specimens deformed at confining pressures less than this were barrelled (traces of conjugate shear zones were visible after deformation at 0.5 kb) but those deformed at higher pressures were more uniformly deformed. These different shapes may be due to end effects which are greater when the specimen increases in volume during deformation.

The volume decreases observed during these experiments must be due to the elimination of porosity since elastic effects are much smaller. It is obvious therefore that the

trend in Fig. III-3, for the volume decreases to be greater with higher confining pressures, must reverse at some finite confining pressure due to the reduction in the pore volume remaining after application of the confining pressure.

During the extension experiments the volume initially increased slightly then decreased before increasing once again at what appeared to be a steady rate but this was not certain due to the lower resolution during extension experiments. The actual scatter during these experiments was less than that during the compression experiments.

Large volume increases occurred during reduction of the confining pressure after deformation in compression (Fig. III-4), the rate of the volume increase increasing as the confining pressure was reduced. The total amount of the volume increase tended to increase with the confining pressure at which the specimen was deformed (see also frontispiece). Considerably smaller volume increases occurred after deformation in extension compared with deformation in compression at similar confining pressures; the volume increase was still large, however, compared with elastic effects indicated by the broken line (taking the compressibility of calcite to be 1.2 megabar^{-1}).

Paterson (1963) observed anomalous length changes after deformation in both compression and extension experiments

using a similar rock, but was unable to distinguish clearly between effects arising from grain distortion and internal cracking.

During compressive deformation at a confining pressure of 8 kb the porosity of the specimens approached zero with no tendency for the volume to increase as the deformation continued which suggests that crystal plasticity was the only mechanism involved. This may also have been the case at 6 kb but the presence of some residual porosity complicates the situation. The volume increases during the later stages of the extension experiments indicate that some cataclasis was involved.

Carrara marble The brittle-ductile transition in compression occurred between 0.5 kb and 1 kb corresponding with the change from a falling to a rising stress-strain curve. The means of the stress-strain curves are drawn in Fig. III-5, the vertical marker indicating the scatter which was about the same at all confining pressures. These curves differ from those for the Solenhöfen limestone in several other respects: no "cross-over" occurs, the stresses are generally lower, and the pressure sensitivity at confining pressures between 6 kb and 10 kb is significantly lower. As with the Solenhöfen limestone, the stress-strain curves for the extension experiments at 6.5 kb had a similar

scatter, but were significantly higher at small strains and had a lower rate of strain hardening compared with the curves for compression experiments at similar confining pressures. These experiments had to be terminated at about 15% strain because the specimens began to "neck".

The volume change curves (Fig. III-6) are similar to those for Solenhöfen limestone, but due to the initially lower porosity, the volume decreases were smaller and the volume increases larger. This trend was also evident in the extension volume change curves (which had less scatter than that indicated by the vertical marker for the compression experiments). Deformation occurred at approximately constant volume in compression with a confining pressure of about 4 kb. As with the Solenhöfen limestone, those specimens which had increased in volume during deformation were barrelled (traces of conjugate shear zones were visible after deformation at a confining pressure of 1 kb) but specimens which had decreased in volume or deformed at constant volume were more uniformly deformed.

Fig. III-7 shows that similar volume increases, though generally smaller ones than those in the Solenhöfen limestone, took place when the confining pressure was reduced after deformation. As with the Solenhöfen limestone the amount of the volume increase after deformation in

compression tended to increase with the confining pressure at which the specimen was deformed (see also frontispiece).

Deformation in compression at confining pressures of 6 kb and 8 kb proceeded with zero porosity and with no tendency for the volume to increase, indicating that crystal plasticity was the only mechanism involved. This conclusion is supported by the low pressure sensitivity at these strains and pressures ($\tan \psi \simeq 0.08$). The equivalent pressure sensitivity for Solenhofen limestone was significantly higher ($\tan \psi \simeq 0.25$) but the reason for this discrepancy is not known. The volume increases during the later stages of the extension experiments indicate that some cataclasis was involved.

Thin sections made of specimens deformed at confining pressures of 1 kb and 10 kb were different in several respects. The 1 kb section showed considerable grain boundary parting and comminution of the individual grains, which were also fairly heavily twinned. The 10 kb section showed no obvious grain boundary parting and little comminution of grains; all grains were saturated with twin lamellae and most of them contained several cracks, but these commonly did not reach a grain boundary and only infrequently crossed an entire grain. No features of the 1 kb section can be unambiguously ascribed to either the

process of deformation or pressure reduction, but in the 10 kb specimen the cracks observed in the specimen presumably occurred during reduction of the confining pressure (if we assume they did not occur during sectioning).

Gosford sandstone After 20% deformation all specimens showed some signs of non-uniform deformation. Specimens deformed at 1 kb confining pressure had broad conjugate shear zones whereas specimens deformed at higher confining pressures were irregularly barrelled but did not display distinct shear zones. Specimens deformed only 10% at 4 kb and 6 kb appeared to be uniformly deformed indicating that the localisation of the deformation was closely associated with the falling sections of the stress-strain curves beyond about 10% strain (Fig. III-8). The pressure sensitivity was high ($\tan \psi \simeq 1.3$) with only a slight decrease at the higher confining pressures.

All specimens decreased in volume at a decreasing rate during the early stages of compression (Fig. III-9) but then increased in volume at a fairly steady rate at a strain which closely corresponded with the downturn of the stress-strain curves.

As with the calcite rocks, the volume increased at an increasing rate as the confining pressure was reduced (Fig. III-10), the total amount of the volume increase

tending to increase with the confining pressure at which the deformation took place (see also frontispiece). The final porosities at atmospheric pressure of all specimens were very similar.

Discussion

These experiments have demonstrated that volume changes which are large compared with elastic effects take place when these three rocks are deformed beyond their elastic range. In order to assess the significance of these volume changes more quantitatively let us consider the basic energy components involved during these experiments and attempt to isolate those due to the volume changes.

Assuming that there are no pore pressure effects and using the notation below with the sign convention defined in Chapter II, i.e. compressive stresses and strains and volume decreases are positive:-

σ_A	Instantaneous axial principal stress.
σ_P	Confining pressure.
$(\sigma_A - \sigma_P)$	Differential stress.
$\delta\varepsilon_A$	Increment of natural axial principal strain.
$\delta\varepsilon_{R1}$, $\delta\varepsilon_{R2}$	Increments of natural radial principal strains.
$\delta v = \delta\varepsilon_A + \delta\varepsilon_{R1} + \delta\varepsilon_{R2}$	Increment of natural volume change (ignoring second order terms).

δW Increment of energy per unit volume transmitted to the specimen.

δW_E Increment of recoverable elastic energy per unit volume transmitted to the specimen.

δW_H Increment of energy dissipated per unit volume as frictional heat.

Balancing the three energy components:-

$$\delta W = \delta W_E + \delta W_H$$

In these experiments changes in recoverable elastic strain energy during deformation were small compared with the energy dissipated as frictional heat. To a first approximation therefore:-

$$\delta W = \delta W_H$$

Now, the work done per unit volume by the differential stress $(\sigma_A - \sigma_P)$ in moving through $\delta\varepsilon_A$ is $\delta\varepsilon_A(\sigma_A - \sigma_P)$ and this is the area under the stress-strain curves if due allowance is made for the difference between natural and conventional strains.

We now define a hypothetical differential stress $(\sigma_A^* - \sigma_P)$ which would be the stress required to cause the same increment of strain $\delta\varepsilon_A$ in the absence of any associated volume change, but using exactly the same mechanisms.

Then:-

$$\delta\varepsilon_A(\sigma_A - \sigma_P) = \delta\varepsilon_A(\sigma_A^* - \sigma_P) - \sigma_P(\delta\varepsilon_A + \delta\varepsilon_{R1} + \delta\varepsilon_{R2})$$

$$= \delta\varepsilon_A(\sigma_A^* - \sigma_P) - \sigma_P\delta v$$

$$= \delta\varepsilon_A\{\sigma_A^* - \sigma_P - \sigma_P(\delta v/\delta\varepsilon_A)\}$$

$$\therefore \quad (\sigma_A - \sigma_A^*) = -\sigma_P(\delta v/\delta\varepsilon_A)$$

and in the limit:-

$$\underline{(\sigma_A - \sigma_A^*) = -\sigma_P(dv/d\varepsilon_A)} \qquad \text{——————} \quad (1)$$

Differentiating -(1) with respect to ε_A:-

$$\underline{\frac{d(\sigma_A - \sigma_A^*)}{d\varepsilon_A} = -\sigma_P(d^2v/d\varepsilon_A^2)} \qquad \text{——————} \quad (2)$$

Thus, the contribution made by any volume change to the strength of the material is equal to the product of the confining pressure and the gradient of the volume change-strain curve. Similarly, the contribution made by the volume change to the strain hardening of the material is equal to the product of the confining pressure and the second derivative of the volume change-strain curve. Volume changes therefore exert a considerable influence on both the level and the shape of the stress-strain curves. It can also be seen that the energy terms due to volume changes

involve all three principal stresses. The intermediate principal stress ranges from the minimum principal stress in the compression experiments to the maximum principal stress in the extension experiments. In any empirical determination of stress-strain properties, the contributions made by any volume changes are automatically included, and correctly so, since they are a real component of the strength under those particular test conditions. It seems likely, however, that the volume changes are dependent on the experimental technique and on the stress path followed, thus giving apparently conflicting results with different techniques. Any general theoretical criterion of deformation should therefore take cognisance of volume changes and of all three principal stresses.

No real material with a Mohr failure envelope (Jaeger, 1962) having a finite slope would be expected to be as strong in extension as it is in compression at the same confining pressure. However if the confining pressure and maximum principal stress at failure during a triaxial compression experiment are σ_P and σ_A respectively and a triaxial extension experiment is then done using the same material and with a confining pressure σ_A it is generally assumed that the minimum principal stress at failure will be σ_P i.e. their Mohr circles are generally assumed to be

coincident. If changes in volume occur during either experiment, however, the resulting Mohr circles will, in general, not be coincident. Even if the volume changes were exactly the same during both experiments the Mohr circles would not be coincident because of the different intermediate principal stresses.

The volume change-strain curves for both Solenhöfen limestone and Carrara marble are distinctly different for compression and extension experiments. This is not surprising since in the former the mean stress is increasing whereas in the latter it is decreasing. The initially higher positions of the extension stress-strain curves compared with those for compression appear to be due to the relative volume increases during the early stages of extension experiments compared with compression experiments but the results are not sufficiently accurate to prove this. The lower rate of strain hardening during the later stages of the extension experiments may be due partly to the second derivative of the volume change-strain curves, but once again it is not possible to make anything other than qualitative statements in this respect. A significant factor in the relative strain hardening rates is undoubtedly the non-uniformity of deformation: any tendency towards a barrel shape during compression experiments will increase

the gradients of these stress-strain curves (which have been calculated assuming uniform deformation) above the true strain hardening rate, and similarly any necking during extension experiments will decrease the gradients to less than the true strain hardening rate.

There does not seem to be any direct relationship between the brittle-ductile transition and the volume change curves, but there is a correlation between ductility and negative values for $d^2v/d\varepsilon_A^2$ which cause a strain hardening effect.

The confining pressure at which constant volume deformation takes place is affected very strongly by the initial porosity of the specimen. In the absence of any volume change measurements during deformation, the general shape of the specimen may give some indication of the sign of the volume change, end effects being more pronounced when the volume is increasing during deformation.

A multiplicity of slip systems has been observed after deformation at high confining pressure at room temperature in calcite, and five independent slip systems may be chosen in any one of several ways (Paterson, 1969). From these experiments confining pressures of at least 6 kb are required to suppress cataclasis during compressive deformation of Solenhöfen limestone and Carrara marble.

Deformation of Gosford sandstone is unlikely to cause significant amounts of slip in the quartz or feldspar crystals, but this does not rule out the possibility of slip occurring in the clay and mica matrix. This may have contributed to the small reduction in the pressure sensitivity with increasing confining pressure.

The volume increases occurring during reduction of the confining pressure after deformation are not the reverse of the volume decreases which occurred as the confining pressure was increased before deformation. The main difference lies in the large accelerating volume increases which occur during the later stages of pressure reduction after deformation in compression. Volume increases occurring after deformation in extension appear to be considerably smaller, but in fact are almost the same as the volume increases occurring after deformation in compression under conditions of the same differential stress if a small allowance is made for the larger deformation during compression experiments (about 20% compared with about 14% during extension experiments). The reasons for these volume increases must lie with internal stresses arising from the elastic and plastic anisotropy of the crystals. Paterson (1963) discussed such stresses in connection with secondary length increases during reduction of the confining pressure after an

experiment, and concluded that the length increases were due to grain distortion and the opening of internal fractures. Such processes are likely to be highly anisotropic and it is not possible to clearly assess their relative importance. No secondary length changes were observed in these experiments, however, which could not be attributed solely to the opening of internal fractures.

If the total volume increase which occurred as the confining pressure was reduced is plotted against the confining pressure at which the specimen was deformed (see frontispiece) an inflection is observed for the Solenhöfen limestone and Carrara marble at a confining pressure of about 2 kb. The reason for this is not known but it may represent the confining pressure at which another family of slip systems is activated.

The magnitude of the volume increases with decreasing confining pressure is such that considerable disturbance of the specimen fabric, associated with internal cracking and opening of pores, is inevitable. Subsequent examination of the specimens or thin sections could therefore easily lead to false conclusions being drawn about the mechanisms of deformation. For example, Orowan (1960 p. 326) stated that the presence of cracks in Carrara marble after deformation in compression at confining pressures up to about

3 kb was evidence for cataclastic deformation. My own experiments with Carrara marble suggest that Orowan arrived at the correct conclusion, though apparently for the wrong reason!

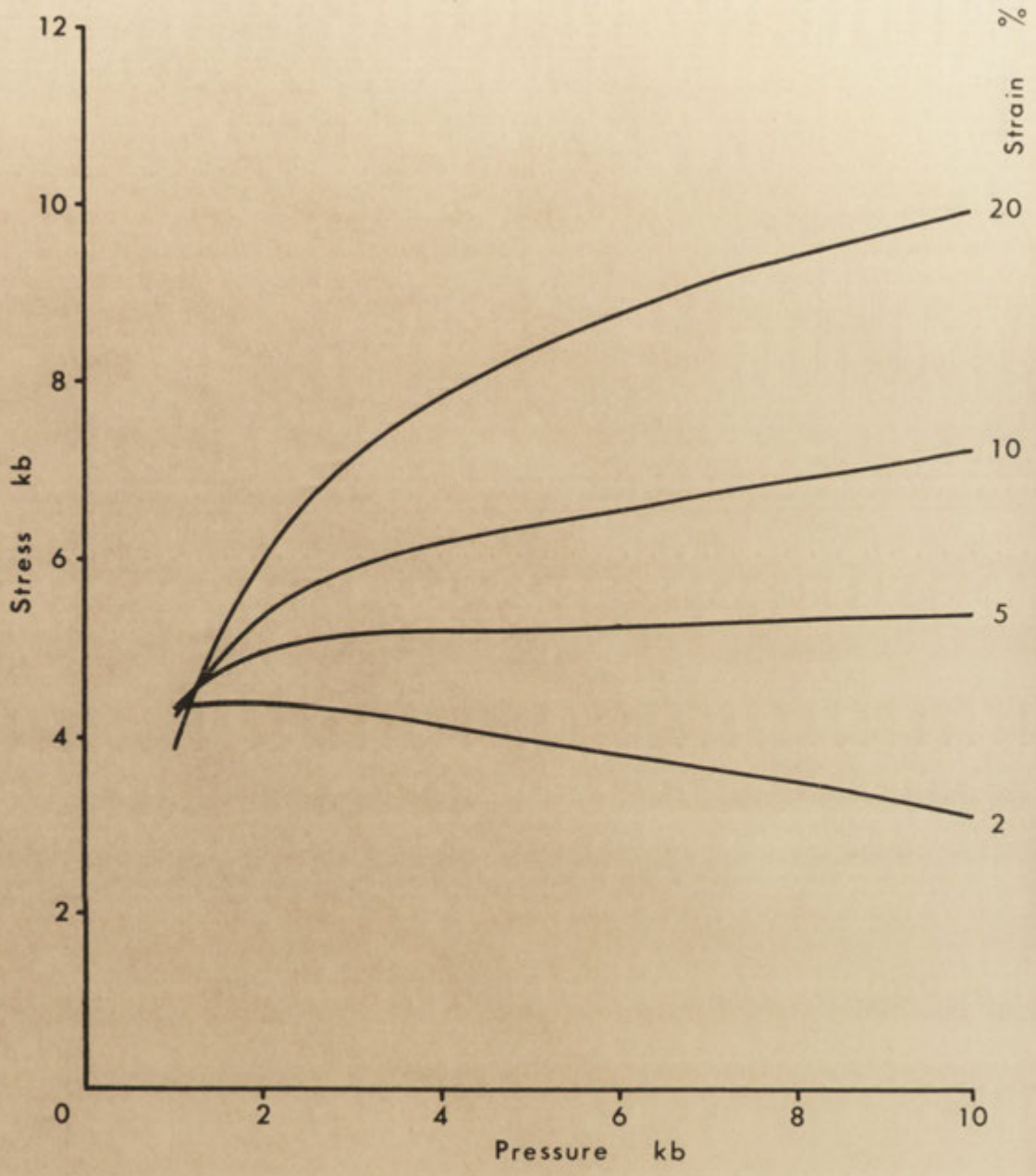


Fig. III-2 Pressure sensitivity curves for Solenhofen limestone.

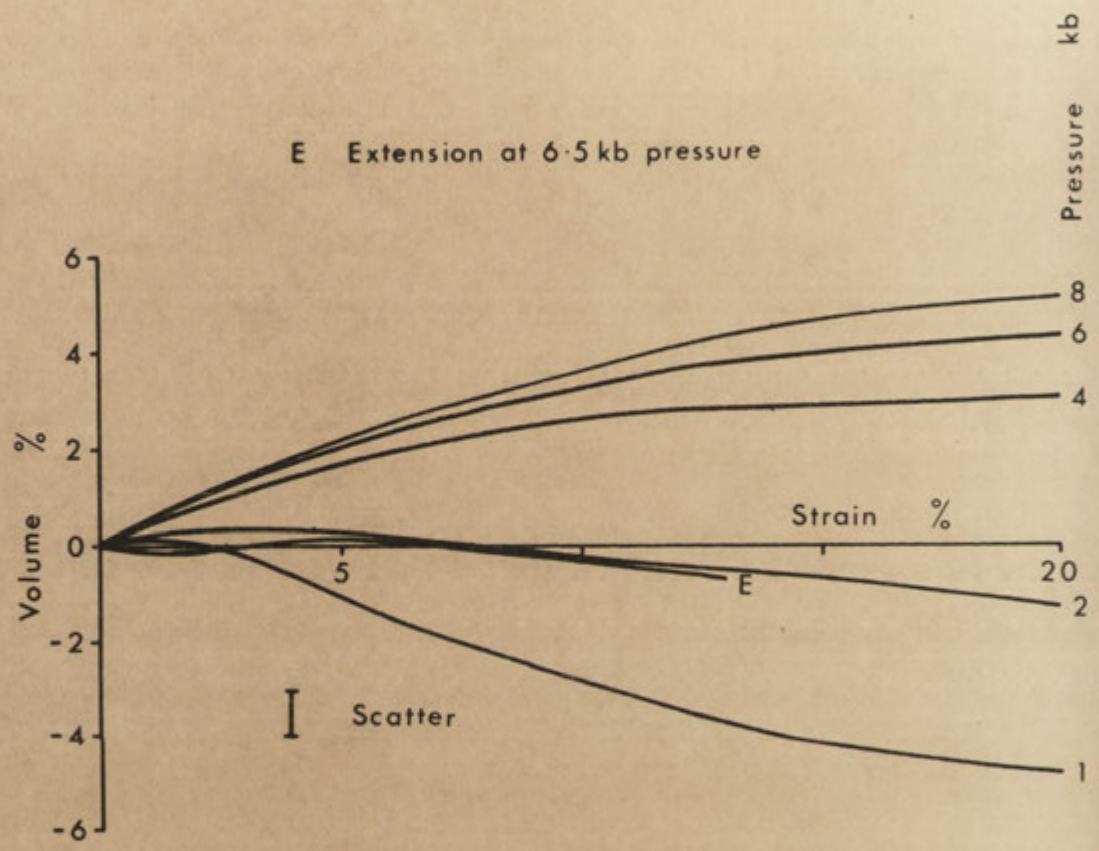


Fig. III-3 Volume change-strain curves for Solenhofen limestone.

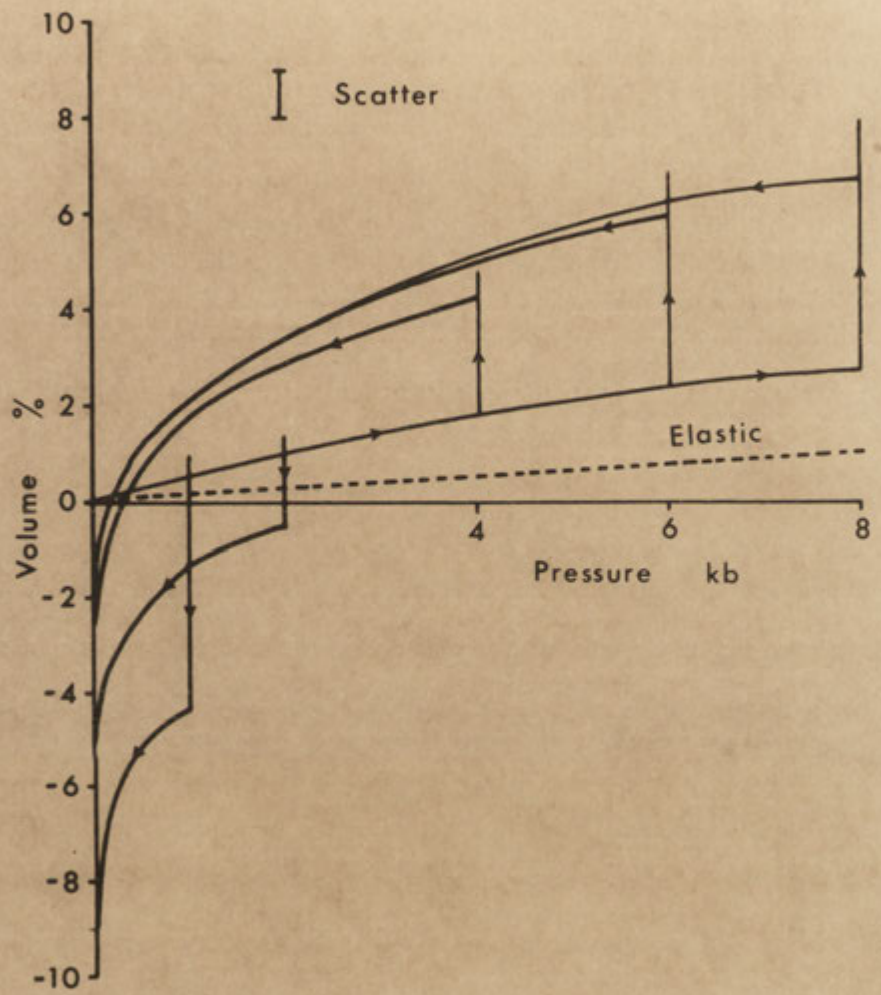


Fig. III-4 Overall volume changes for Solenhöfen limestone.

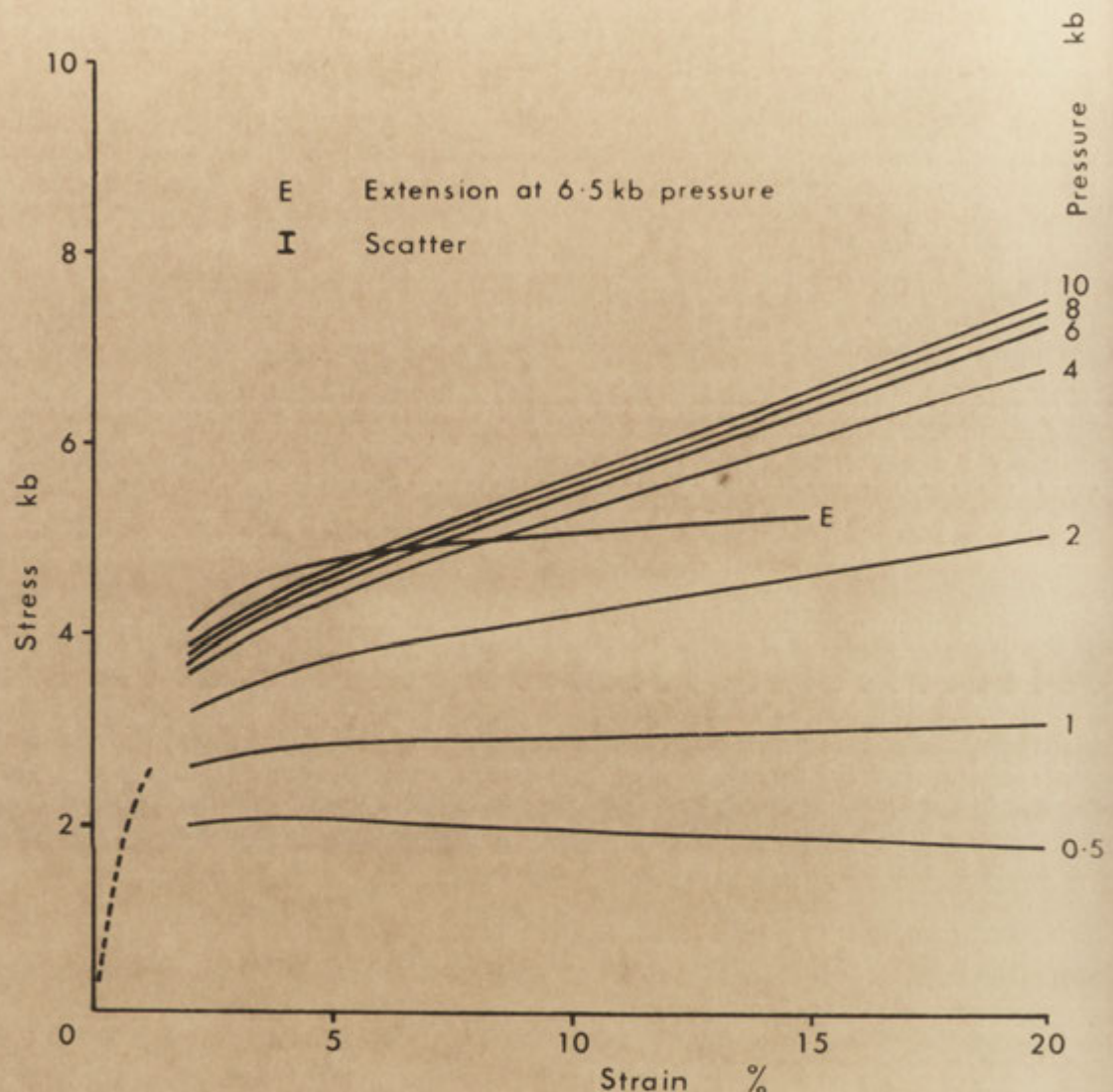


Fig. III-5 Stress-strain curves for Carrara marble.

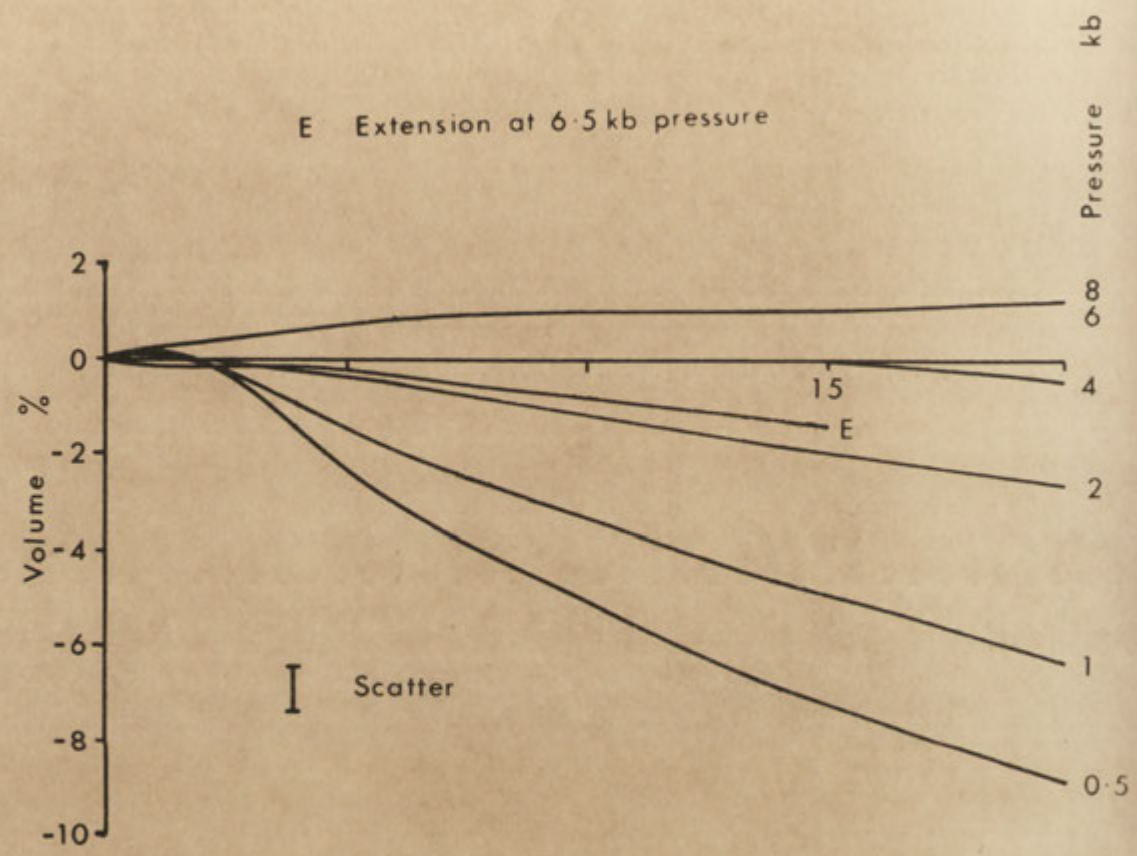


Fig. III-6 Volume change-strain curves for Carrara marble.

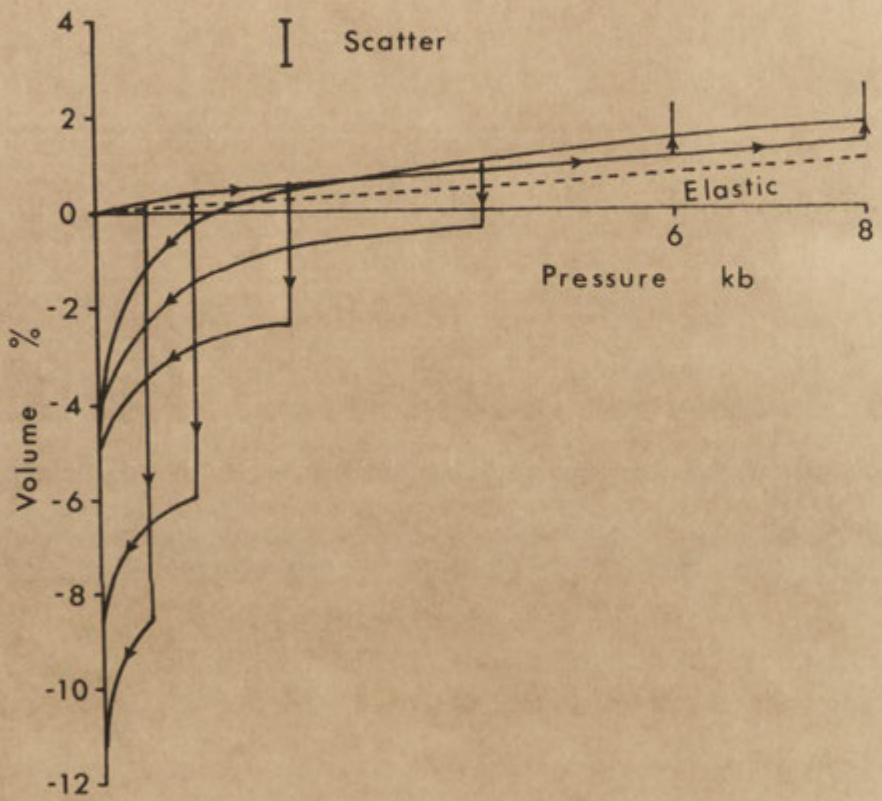


Fig. III-7 Overall volume changes for Carrara marble.

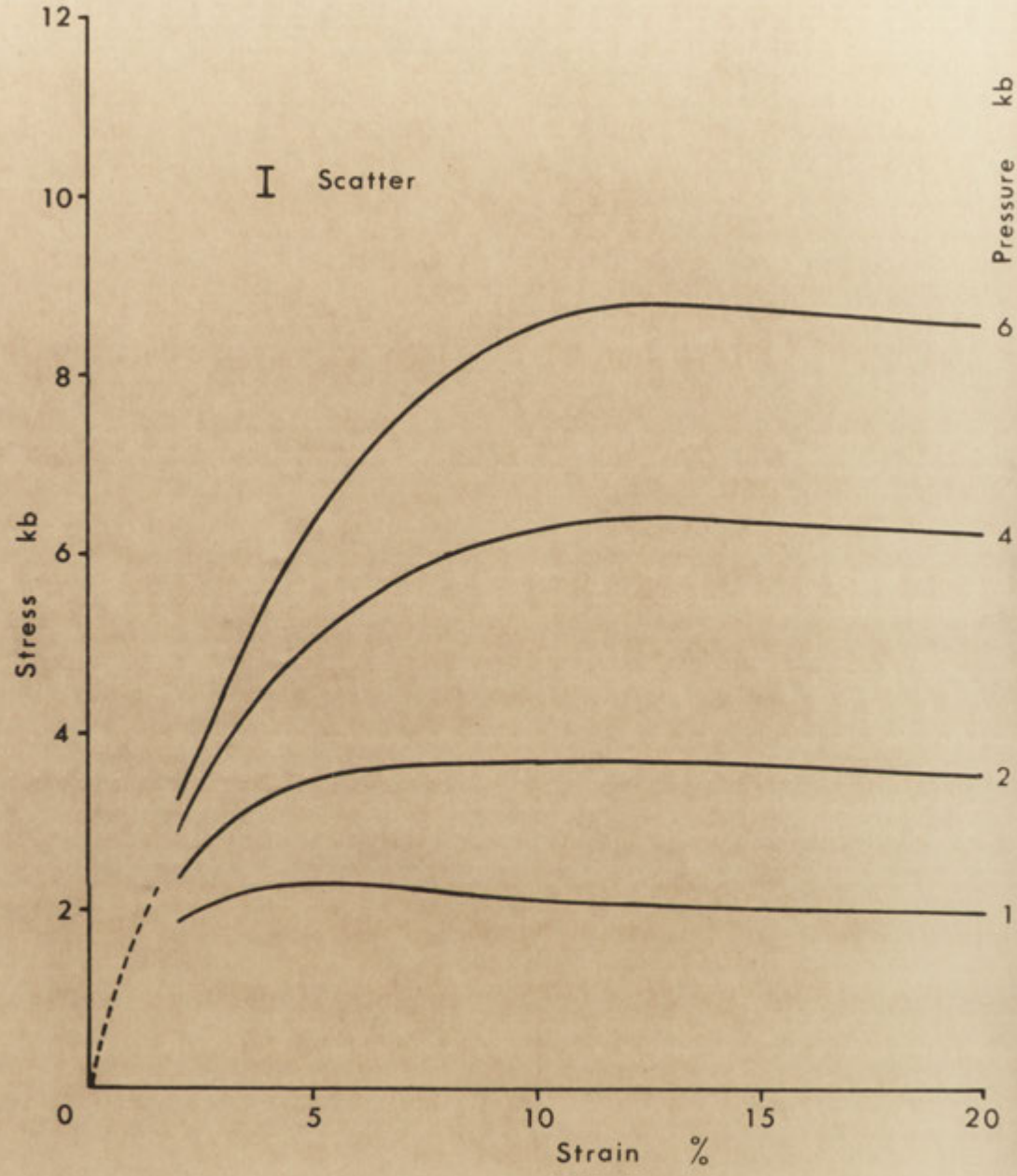


Fig. III-8 Stress-strain curves for Gosford sandstone.

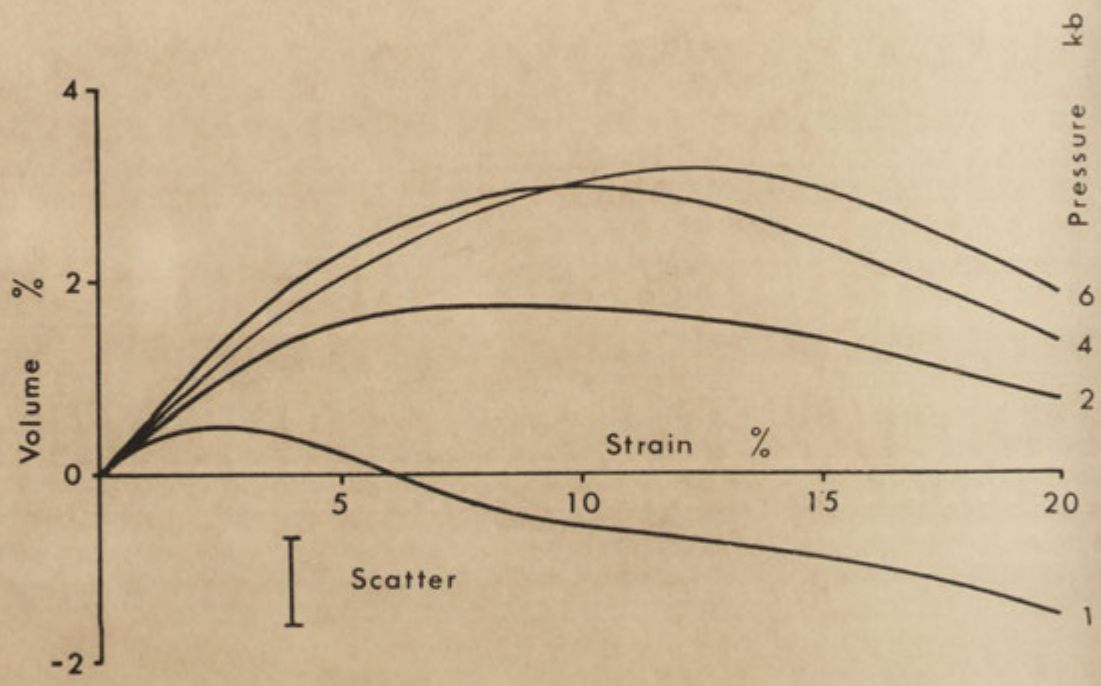


Fig. III-9 Volume change-strain curves for Gosford sandstone.

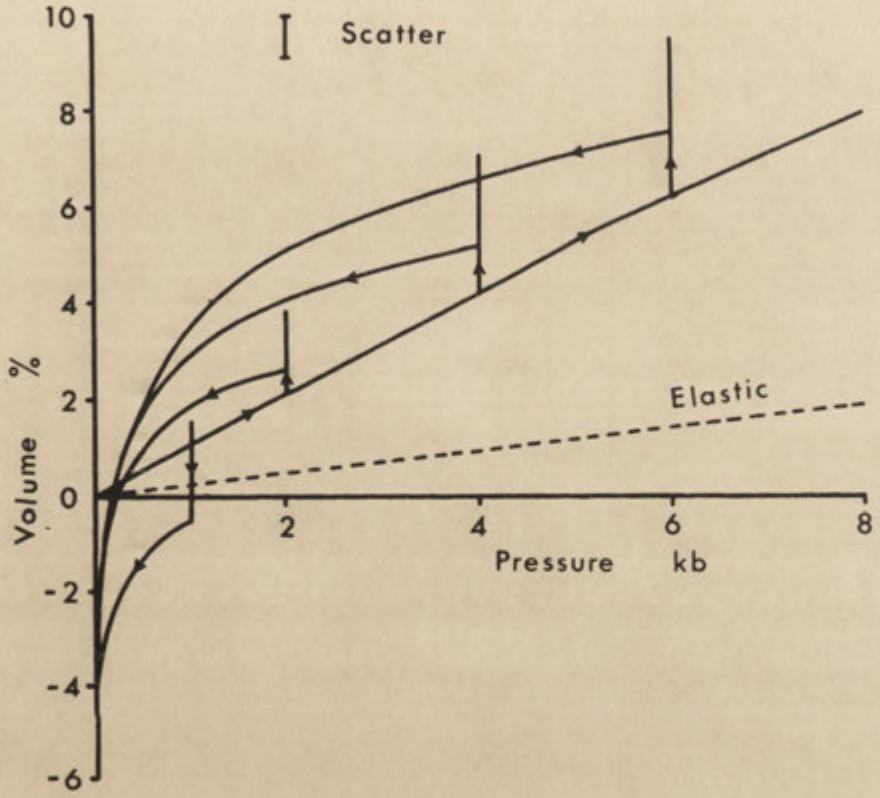


Fig. III-10 Overall volume changes for Gosford sandstone.

CHAPTER IV

DEFORMATION

OF

TALC AT ROOM TEMPERATURE

Introduction

These experiments were done for two main reasons. Firstly, talc is the most commonly used material in solid medium pressure apparatuses (see Chapter IX) yet very little published information existed on its stress-strain properties particularly at high confining pressures. Secondly, the results are of more general interest on account of what they may contribute to a better understanding of the mechanisms of deformation in those materials which have a platy structure and which are thought to have only one slip plane.

Materials

All specimens were made by grinding 10 mm diamond drill cores to lengths of 20 mm.

Three Springs talc (Western Australia). Block A was white and consisted of a felted mass of minute crystals of talc threaded by veins and irregular patches consisting of coarser crystals of talc. A trace of sphene and iron ore minerals were also present. In addition to thin section examination, X-ray texture measurements revealed no preferred orientation of the talc, and no anisotropy was detected on the stress-strain curves by deforming three specimens cored in orthogonal directions at a confining pressure of 4 kb. The bulk density was 2.71 g. cm^{-3}

giving a theoretical porosity of about 3.2%. After air drying at room temperature for several weeks the specimens were found to still contain up to about 0.4% by weight of water if placed in an oven at 105°C for several hours. Similar amounts of water were found in talc which had not been wet during the drilling process. Specimens used in these experiments had not been oven dried.

Block B was pale green and consisted of slightly finer talc crystals than block A and no preferred orientation was apparent in thin section. A trace of spinel was present and some narrow veins of serpentine cut the rock; any specimen which intersected one of these veins was rejected. The bulk density was 2.75 g. cm^{-3} giving a theoretical porosity of about 1.8%.

Griggs' talc. Three small white blocks (C, D and E) were supplied by Professor D. Griggs from different batches of commercially obtained material which were used in solid medium pressure apparatuses in his laboratory. Examination of thin sections revealed conspicuous preferred orientations of the talc crystals in all blocks.

Techniques

Three Springs talc, block A Room temperature volume change compression experiments as described in Chapter II. Three specimens were deformed at each of the confining pressures

2 kb, 4 kb, 6 kb and 8 kb. Three specimens were also deformed at a confining pressure of 10 kb in room temperature non volume change compression experiments as described in Chapter II. Annealed copper jackets with a wall thickness of 0.25 mm were used for these experiments.

Three Springs talc, block B Room temperature non volume change compression experiments as described in Chapter II. Two specimens of arbitrary orientation were deformed at a confining pressure of 6 kb. Annealed copper jackets with a wall thickness of 0.25 mm were used.

Griggs' talc Room temperature non volume change compression experiments as described in Chapter II. Two pairs of mutually perpendicular specimens were cored from each block and deformed at a confining pressure of 6 kb. The symbols $\parallel$ (parallel), and $\perp$ (perpendicular) after the block letter refer to the approximate relative orientations of the specimen axis and the plane in which the talc basal sections preferred to lie. Annealed copper jackets with a wall thickness of 0.25 mm were used.

Results

The means of the stress-strain curves for the Three Springs block A talc at each confining pressure are drawn in Fig. IV-1; the vertical marker indicates the scatter which was similar at all confining pressures. The means of

the stress-strain curves for blocks B, C and E are drawn in Fig. IV-2; the vertical marker indicates the typical scatter between duplicate experiments. The stress-strain curves for D$\parallel$ and D$\perp$ were very similar to those for C$\parallel$ and C$\perp$ and have been omitted in the interests of clarity.

Specimens deformed at a confining pressure of 2 kb showed signs of broad conjugate shear zones and had steadily falling stress-strain curves. At 4 kb the specimens were irregularly barrelled and had fairly level stress-strain curves. At 6 kb, 8 kb and 10 kb the Three Springs block A talc specimens were fairly uniformly deformed and had continually rising stress-strain curves. It can be seen that the levels of the stress-strain curves rise with increasing pressure, but at a decreasing rate. The Three Springs block B talc specimens deformed at a confining pressure of 6 kb showed signs of broad conjugate shear zones and had rather undulating stress-strain curves which were generally higher than the equivalent block A curves. Specimens from blocks C, D and E deformed at a confining pressure of 6 kb were fairly uniformly deformed and had continually rising stress-strain curves but the cross sections of the specimens were noticeably oval.

During the volume change experiments, all Three Springs block A talc specimens initially decreased slightly in

would have five independent slip systems. Paterson emphasised however, that under no circumstances does kinking permit the body to be extended parallel to the slip plane or shortened normal to the slip plane. In this respect kinking is analogous to some types of twinning.

There are only five independent components of strain during a general uniform deformation if a constant volume restriction is imposed and one independent slip system can intuitively be associated with each of them. Each of these five independent components of strain must be capable of taking on either positive or negative values during general uniform deformation. Alternatively, we can consider them to be ten "independent half components" of strain if each of these half components can only take on one sign. One can intuitively associate an "independent half slip system" with each of these ten "independent half components" of strain.

Now, considering the additional equivalent independent slip systems postulated by Paterson, we see that two of the ten "independent half components" of strain cannot exist because the body cannot be extended parallel to the slip plane (it is also impossible for the body to be shortened normal to the slip plane, but this is already covered by the no volume change requirement and does not cause any

further restriction). Only eight of the ten "independent half components" of strain can therefore be satisfied even with three non-parallel slip directions on a unique slip plane and with all the kinking systems interpenetrating. If each of these eight "independent half components" of strain are associated with an "independent half slip system" we see that only four fully independent slip systems can intuitively exist under these conditions.

It is possible that this number would be sufficient to satisfy the requirement for a polycrystalline body to undergo arbitrary deformation if the restriction requiring homogeneous strain on the scale of the crystals throughout the body is removed. This restriction is not adhered to in practice; several workers have demonstrated heterogeneous strain on the scale of the crystals in metals (eg. Carpenter and Elam, 1921; Boas and Hargreaves, 1948; Urie and Wain, 1952-1953).

It may be that crystals which are oriented in such a way that it is impossible for them to deform by intra-crystalline plasticity behave like rigid inclusions in a flowing matrix if the confining pressure is sufficiently high to prevent cataclasis; their share of the deformation being provided by neighbouring crystals which lie in more favourable orientations. Such a process would be likely to

disturb the orientation of the original crystal and cause it to become more favourably oriented so that it would then contribute to the deformation.

In principle therefore it seems possible for polycrystalline talc to deform arbitrarily by means of intra crystalline plasticity using only the basal plane for slip. It is probably more likely, however, that some non-basal slip systems become activated under extreme conditions, but there is no direct evidence at the present time to prove that these exist.

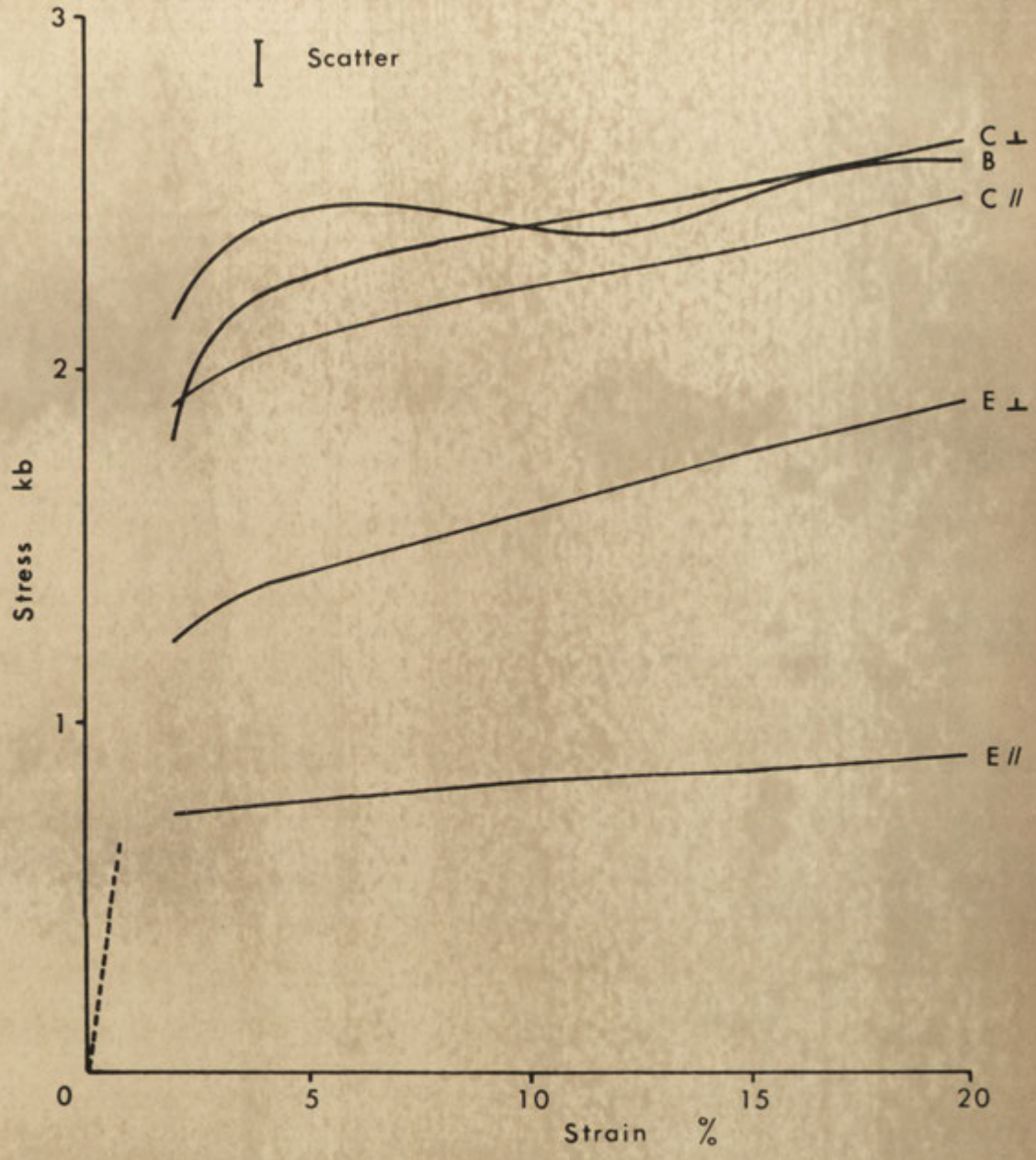


Fig. IV-2 Stress-strain curves for various talcs at a confining pressure of 6 kb.

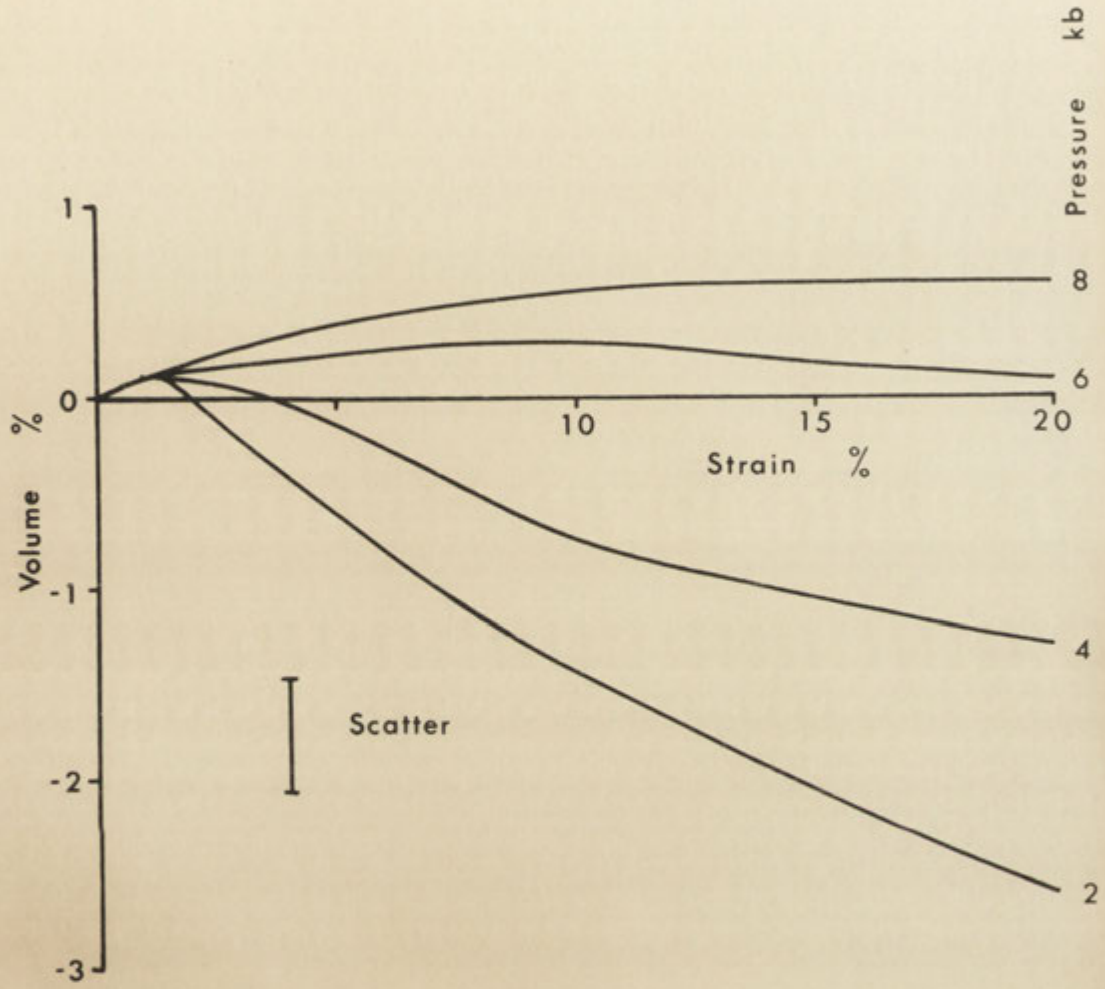


Fig. IV-3 Volume change-strain curve for Three Springs talc.

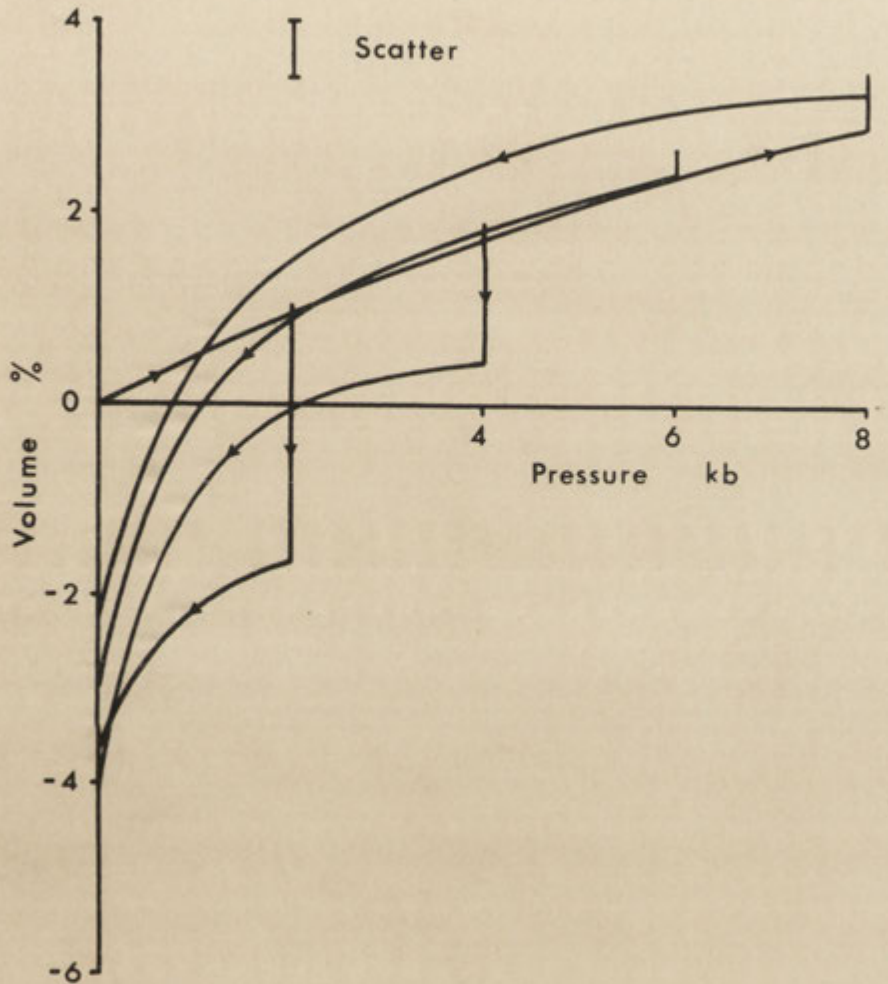


Fig. IV-4 Overall volume changes for Three Springs talc.

CHAPTER V

DEFORMATION

OF

TALC AT HIGH TEMPERATURE

Introduction

These experiments were done mainly to expand on Chapter IV, and to determine how talc is likely to behave when used in solid medium pressure apparatuses at high temperatures (see also Chapter IX).

The work of Kitahara, Takenouchi and Kennedy (1966) showed that the decomposition temperature for talc increases from approximately 750°C to 850°C as the confining pressure increases from atmospheric pressure to 30 kb. At 4 kb, the confining pressure chosen for our experiments, the decomposition temperature is approximately 800°C.

The decomposition process can be written:-

$$Mg_3Si_4O_{10}(OH)_2 \rightarrow 3MgSiO_3 + SiO_2 + H_2O$$

i.e. TALC → ENSTATITE + QUARTZ + WATER

Materials

The Three Springs talc (Block A) specimens used in these experiments were taken from the same batch as those deformed at room temperature. The specimens had been air dried for several weeks at room temperature after coring but were found subsequently to still contain up to 0.4% by weight of water if oven dried at 105°C for several hours. Similar amounts of water were found in

talc which had not been wet during the drilling process. Since it had not been the practice to oven dry the talc before using it in the solid medium apparatus, all the specimens were used in the air dried condition except for two oven dried specimens deformed at 600°C.

Techniques

The high temperature pressure apparatus, venting arrangement and specimen jacketing are described in Chapter II. The confining pressure was raised to 4 kb before increasing the temperature which was maintained constant at the required level for approximately 30 minutes before deforming the specimen about 10% at a strain rate of $4 \times 10^{-4} sec^{-1}$. After deformation the pressure and the temperature were reduced simultaneously. The process of increasing and decreasing the temperature took up to about 10 minutes.

Samples were taken from some of the deformed specimens and X-rayed using the X-ray diffractometer.

Results

200°C Almost the same as at room temperature with a confining pressure of 4 kb. The three specimens were slightly discoloured (grey) and the levels of the stress-strain curves were slightly lower. No differences were observed between vented and unvented specimens.

300°C (Unvented only) The two specimens showed signs of broad conjugate shear zones and were discoloured. The slopes of the stress-strain curves were the same as at the lower temperatures but their general levels were slightly lower than those at 200°C.

400°C All specimens were discoloured. The one vented specimen showed signs of a shear plane but no sharp drop occurred in the stress-strain curve which was at about the same level as those at 300°C. The two unvented specimens failed on shear planes, sharp drops occurring at about 2% strain on their stress-strain curves. The general levels of the stress-strain curves of the unvented specimens were considerably lower than that of the vented specimen.

500°C (Unvented only) All three specimens were strongly discoloured and failed on shear planes, sharp drops occurring at about 2% strain on the stress-strain curves which were generally similar to those for unvented specimens at 400°C.

600°C All specimens were strongly discoloured and failed on shear planes, sharp drops occurring at about 2% strain on all the stress-strain curves. The general levels of the stress-strain curves for the air dried specimens were lower than those at 500°C, the curves for the two unvented specimens being lower than that for the vented specimen. The curves for the two oven dried specimens (also vented)

were significantly higher than those for the air dried specimens.

700°C Both specimens were strongly discoloured. The vented specimen deformed fairly uniformly and no stress drop occurred on the stress-strain curve. The unvented specimen failed on a shear plane with an associated stress drop at about 4% strain. The general levels of both stress-strain curves were lower than that of the corresponding curve at 600°C.

800°C All specimens were slightly discoloured. The two vented specimens deformed fairly uniformly and no stress drop occurred on the stress-strain curves. The unvented specimen failed on a shear plane with an associated stress drop at about 4% strain. The general levels of these curves were similar to those at 700°C.

900°C None of these specimens was discoloured, but they were very hard when examined and could not be scratched with a penknife blade. The two vented specimens deformed fairly uniformly but the unvented specimen showed signs of a shear plane. None of the stress-strain curves showed stress drops and were at the same general level as those at 700°C and 800°C.

The maximum differential stress attained during each of these experiments is plotted against temperature in Fig. V-1.

X-ray diffractometer records of powdered material taken from deformed specimens showed that no decomposition of talc occurred except during experiments at 900°C. Some breakdown of the talc had been anticipated at 800°C, but its absence could be due to either small errors in the temperature calibration or to the relatively short time spent at that temperature. After experiments at 900°C significant quantities of talc still remained in addition to quartz, enstatite and anthophyllite, $(Mg,Fe)_7Si_8O_{22}(OH)_2$. The presence of anthophyllite was indicated by peaks corresponding to spacings of 8.30 Å, 3.66 Å, 3.06 Å, 2.75 Å, 1.73 Å and 1.62 Å. Other anthophyllite peaks would have been indistinguishable from peaks due to other components.

Discussion

The presence of adsorbed water in these specimens had an important influence on their behaviour at high temperatures. Driving off this adsorbed water may be associated with the change in colour of the specimens from white to grey, but the reason for this is not known. We can calculate the approximate pore pressures caused by this water if it does not escape, assuming 0.016 g of water in each specimen and an initial porosity of about 3.2% at atmospheric pressure and room temperature. Fig. IV-4 indicates that the porosity of the specimen would be reduced to about 1.5% when the

confining pressure is increased to 4 kb. We will now assume that this porosity remains unchanged as the temperature is increased. This assumption is probably justified for temperatures up to about 400°C, but some reduction of the porosity may occur at higher temperatures.

Bulk volume of specimen = 1.56 cm^3

Pore volume due to 1.5% porosity = 0.0234 cm^3

Maximum specific volume of water = 1.46 $cm^3 \cdot g^{-1}$

The pressure of this water at various temperatures (Kennedy and Holser, 1966) will be as follows. The effective confining pressure is the actual confining pressure reduced by an amount equal to the pore pressure.

Temperature	Pore pressure	Effective pressure
300°C	0.1 kb	3.9 kb
400°C	0.9 kb	3.1 kb
500°C	1.9 kb	2.1 kb
600°C	2.8 kb	1.2 kb

At temperatures of 700°C and above the effective confining pressure would be expected to be zero. During deformation the volume of the specimen may be expected to vary in a similar manner to specimens at room temperature with a confining pressure equal to the effective confining pressure in the high temperature experiments provided that the temperature is not too high. Fig. IV-3 indicates that

is it clear why the oven dried specimens still failed in a brittle manner. The flow stress of the air dried vented specimens is apparently lower in most cases than the differential stress required to cause a shear plane type of failure. The higher pore pressures in the unvented specimens however reduce the effective confining pressure and so reduce the differential stress required to cause a shear plane type failure to less than the flow stress. The unvented specimens should therefore have lower maximum differential stresses than those for vented specimens at the same temperature. This difference need not be a large one, however, and appears in this case to be less than the experimental scatter. The oven dried vented specimens deformed at 600°C should have lower pore pressures than the air dried vented specimens deformed at the same temperature and should therefore be more likely to deform in a ductile manner. The flow stress for the oven dried specimens appears to be significantly higher than that for the air dried specimens, however, and the differential stress required for a shear plane type of failure is attained first.

During experiments at 900°C more water becomes available as a result of the decomposition of the talc, but even in the unvented specimens the flow stress appears to be less

CHAPTER VI

DEFORMATION
OF
A TALC-TREMOLITE ROCK

Introduction

After having studied the effects of pressure on the deformation of relatively pure talc rocks it is of interest to compare these results with results from similar experiments using a talc rock containing a second mineral component. This rock is of additional interest because of its triaxial or orthorhombic anisotropy resulting from strong preferred orientations of its two mineral components.

Materials

The origin of this rock is not known.

The rock is schistose with needles of tremolite, some of which are about 5 mm long, lying more or less parallel to the schistosity and embedded in a mass of talc. The tremolite needles also have a preferred lineation within the plane of the schistosity, the s-surface. The flakes of talc commonly lie parallel to the s-surface but their preferred orientation is not as well defined as that of the tremolite . The talc and the tremolite are in about a 3:1 ratio and the rock has a bulk density of 2.85 $g.cm^{-3}$.

The preferred orientation of the tremolite has been used to define three orthogonal axes as in Fig. VI-1. The rotating arrows on the vertical and approximate clino axes indicate the most common deviations from this position.

Techniques

10 mm diameter specimens were cored in the X, Y and Z directions and also in a direction at 45° to both Y and Z. The specimens were ground to a length of 20 mm.

Red rubber jackets with a wall thickness of 1.6 mm were used for non volume change room temperature compression experiments at confining pressures of 0.5 kb, 1 kb, 2 kb, 4 kb and 8 kb. Three experiments were done at each combination of these confining pressures and specimen orientations.

Results

Sets of stress-strain curves have been drawn for each specimen orientation (Figs. VI-2, 3, 4, 5). The inset diagram on each of these figures illustrates the preferred orientations of the tremolite and talc crystals for that specimen orientation. The broken lines indicate the flow or failure surfaces as deduced from the ovality of cross section of the specimens after deformation.

X orientation At a confining pressure of 0.5 kb specimens failed on single slickensided shear planes inclined at about 40° to the differential stress direction. At 1 kb deformation was by shearing in narrow conjugate shear zones, the specimens remaining intact. Wider conjugate shear zones were formed at 2 kb and 4 kb and the deformation was distributed through the specimen at 8 kb.

In every case the cross section of the specimen was oval with the long axis of ovality in the Y direction.

The means of the stress-strain curves for this orientation are shown in Fig. VI-2, the vertical marker indicating the scatter of the original curves.

Y orientation At a confining pressure of 0.5 kb specimens failed on single slickensided shear planes inclined at about 40° to the differential stress direction. At 1 kb deformation was by shearing in narrow single or conjugate shear zones, the specimens remaining intact. With increasing confining pressure the deformation became more distributed until at 8 kb no shear zones were visible. Several of the specimens deformed at 0.5 kb, 1 kb, 2 kb and 4 kb were found to be fractured on planes perpendicular to the differential stress direction, presumably due to the deformation causing a breakdown of cohesion across the s-surface. In every case the cross section of the deformed specimen was oval with the long axis of ovality in the X direction.

The means of the stress-strain curves for this orientation are shown in Fig. VI-3.

Z orientation At a confining pressure of 0.5 kb two of the specimens failed on a single slickensided shear plane and one deformed on an intersecting kink band and shear plane. At the higher confining pressures specimens deformed

by bending and with kink bands which became wider and sometimes conjugate with increasing pressure. Shear planes were inclined at about 30° to the differential stress direction but the kink bands were inclined at about 45°. After deformation at 4 kb and particularly at 8 kb kink bands and bending were observed in individual tremolite crystals. In every case the cross section of the deformed specimen was oval with the long axis of ovality in the Y direction.

The means of the stress-strain curves for this orientation are showed in Fig. VI-4.

45YZ orientation At a confining pressure of 0.5 kb specimens failed on single slickensided shear planes which were parallel to the s-surface. As the confining pressure was increased the shear zone became more evenly distributed throughout the specimen but remained parallel to the s-surface. Shear deformation of this type requires either that the ends of the specimen become offset or that rotation of the shear planes should take place. In these experiments the latter alternative occurred and this resulted in the s-surface being rotated at the centre of the specimen to about 55° to the differential stress direction.

The means of the stress-strain curves for this orientation are shown in Fig. VI-5.

Discussion

For all orientations increasing the confining pressure significantly increased the flow stress of the rock. The sensitivity of the flow stress to changes of confining pressure increased with increasing strain but decreased with increasing confining pressure. This suggests that the amount of cataclastic deformation is generally reduced at higher confining pressures.

There was no unique strongest or weakest orientation, the pressure sensitivities and rates of strain hardening varying between the four different orientations. This may be compared with the results of work that has been done on the brittle failure of anisotropic rocks by several workers such as Donath (1961,1964), Hoek (1964) and Borg and Handin (1966) who found that the stress at failure was least when the plane of the foliation was inclined at an angle of 30° to 45° to the differential compressive stress direction. The Z orientation was the strongest except after 20% strain at 8 kb when the Y orientation was stronger. The X orientation was the weakest except at about 1% strain when the 45YZ orientation was weaker.

For orientations X, Y and 45YZ it can be seen from the preferred orientation diagrams in Figs. VI-2, 3, 5 respectively that deformation has proceeded in a manner which

least involved the tremolite crystals. It does not seem likely that the tremolite needles would have any effect analogous to that in fibre reinforced materials (eg. Holister and Thomas, 1966) in preventing flow of the talc in the Z direction in X and Y orientation specimens because the tremolite crystals in thin sections of undeformed rock invariably had multiple cracks perpendicular to their vertical axis; they could not therefore be expected to have any extensile strength. With the Z orientation, involvement of the tremolite crystals was inevitable although the tendency towards kinking and bending modes of deformation (on the scale of the specimen) reduced the number of tremolite crystals which had to undergo shear failure. The axis of the bending and kinking of the specimen is probably determined mainly by the preferred orientation of the talc crystals which have a low flexural stiffness about all axes lying within the s-surface. The inequant shape of the tremolite crystals in cross section would also favour this axis.

Without knowing the stress-strain properties of the talc in the absence of the tremolite it is difficult to assess the exact effect of the tremolite. The relative orientation of the talc crystals, however, is identical in specimen orientations X and Z (assuming no preferred

crystallographic direction in the talc cleavage plane) both of which were cored parallel to the s-surface. The considerable differences in the stress-strain properties between these two orientations must therefore be ascribed to the preferred lineation of the tremolite crystals.

The presence of some bending and kink bands in tremolite crystals after deformation of the Z orientation specimens at the higher confining pressures is indicative of at least one slip system operating in tremolite at room temperature. This is the only known evidence for slip in an amphibole except for a recent abstract by Rooney and Riecker (1969). Very large longitudinal stresses (i.e. parallel to the vertical axis of the crystals) can be induced due to effects analogous to those utilised in the technology of fibre reinforced materials (eg. Holister and Thomas, 1966).

Consider a situation similar to that during the early stages of deformation of Z orientation specimens in which we assume that the talc matrix is yielding but the tremolite crystals are still deforming elastically. We further assume that the shear stress at the talc-tremolite interface is half the flow stress, σ_{talc}, of the talc at the confining pressure of the experiment. The differential stress, σ_D, at the middle of a needle of length L, perimeter of cross

section P and cross sectional area A is given approximately by the relation

$$\sigma_D = \sigma_{talc} + \frac{LP\sigma_{talc}}{4A}$$

$$\text{i.e.} \quad \sigma_D = \sigma_{talc}\left(1 + \frac{LP}{4A}\right)$$

When σ_D reaches the flow stress of the tremolite σ_{trem} (we assume that $\sigma_{trem} >> \sigma_{talc}$) for that orientation some relaxation and redistribution of σ_D will occur.

Considerable energy will therefore be stored as elastic strain energy in the Z orientation specimens before the flow stress for the specimen is attained. The flow stress for the Z orientation specimens would therefore be expected to be higher than that for other orientations during the early stages of deformation.

For specimen orientations X, Y and 45YZ, deformation could in theory have taken place with only minimal involvement of the tremolite. Even for these orientations, however, the volume of the tremolite would lead to considerable heterogeneity of deformation in the talc and even the weakest orientations are rather stronger than would have been expected in a pure talc rock with a similar preferred orientation.

The different rates of strain hardening of the different orientations appear to be related to their different degrees of "structural stability". In the Z orientation specimens yielding occurs in a manner analogous to the instability usually arising from the buckling of a column or strut when subjected to its critical (Euler) load. Because of the relatively small length diameter ratio, however, complete failure does not occur except at the lower confining pressures and the specimens retain most of their original load bearing capacity. In contrast both the tremolite and the talc crystals in the Y orientation specimens are in their most stable orientations and rates of strain hardening during distributed deformation are relatively high. X orientation specimens have a structure which is intermediate in stability between that of the Y and Z orientation specimens and they have a corresponding intermediate rate of strain hardening during distributed deformation. The 45YZ specimens display strain hardening properties during distributed deformation due to the rotation of the s-surface from the original 45° to the direction of the differential stress to considerably larger angles. The structure therefore tends towards that of the Y orientation specimens.

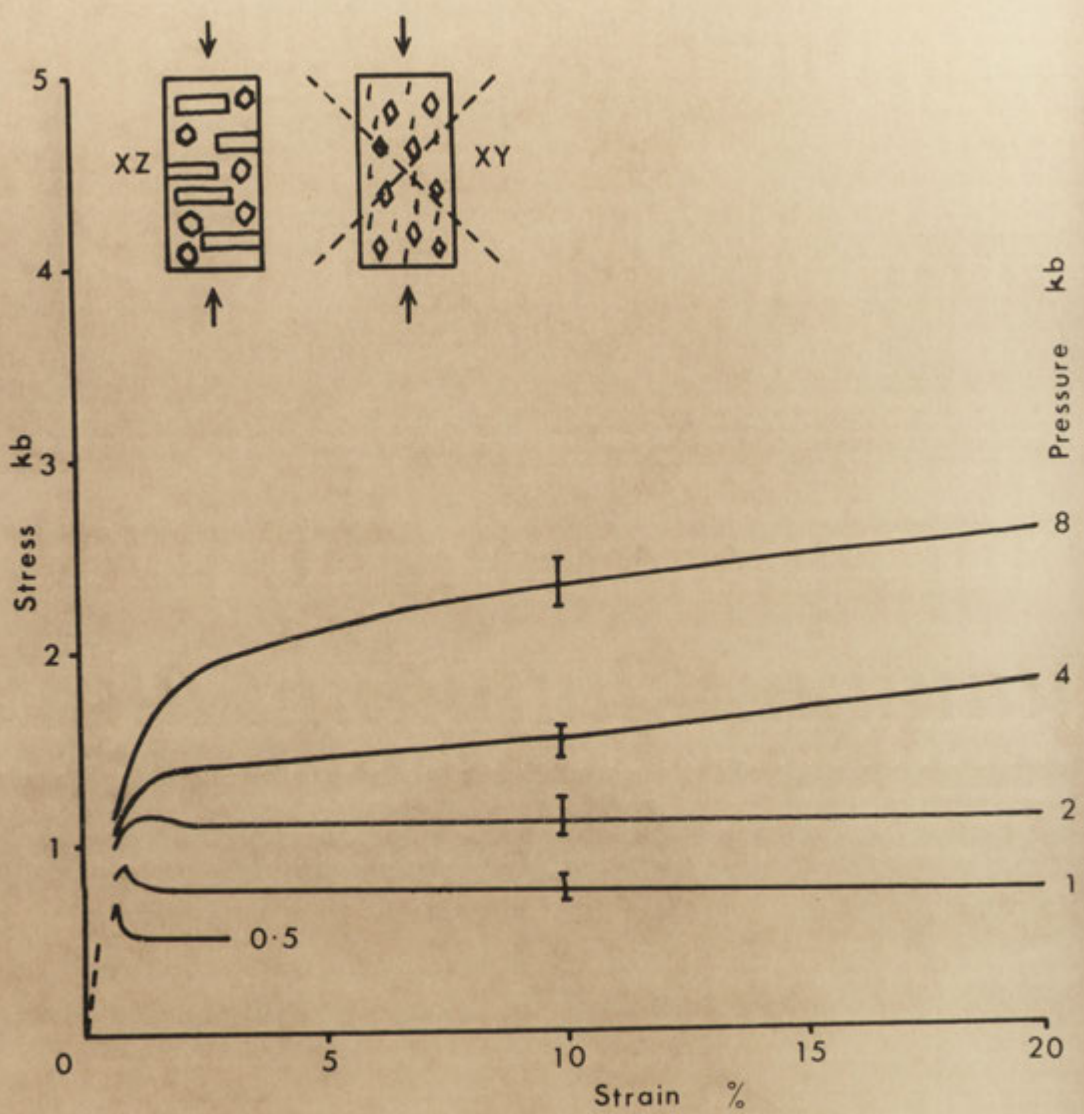


Fig. VI-2 Stress-strain curves for X orientation.

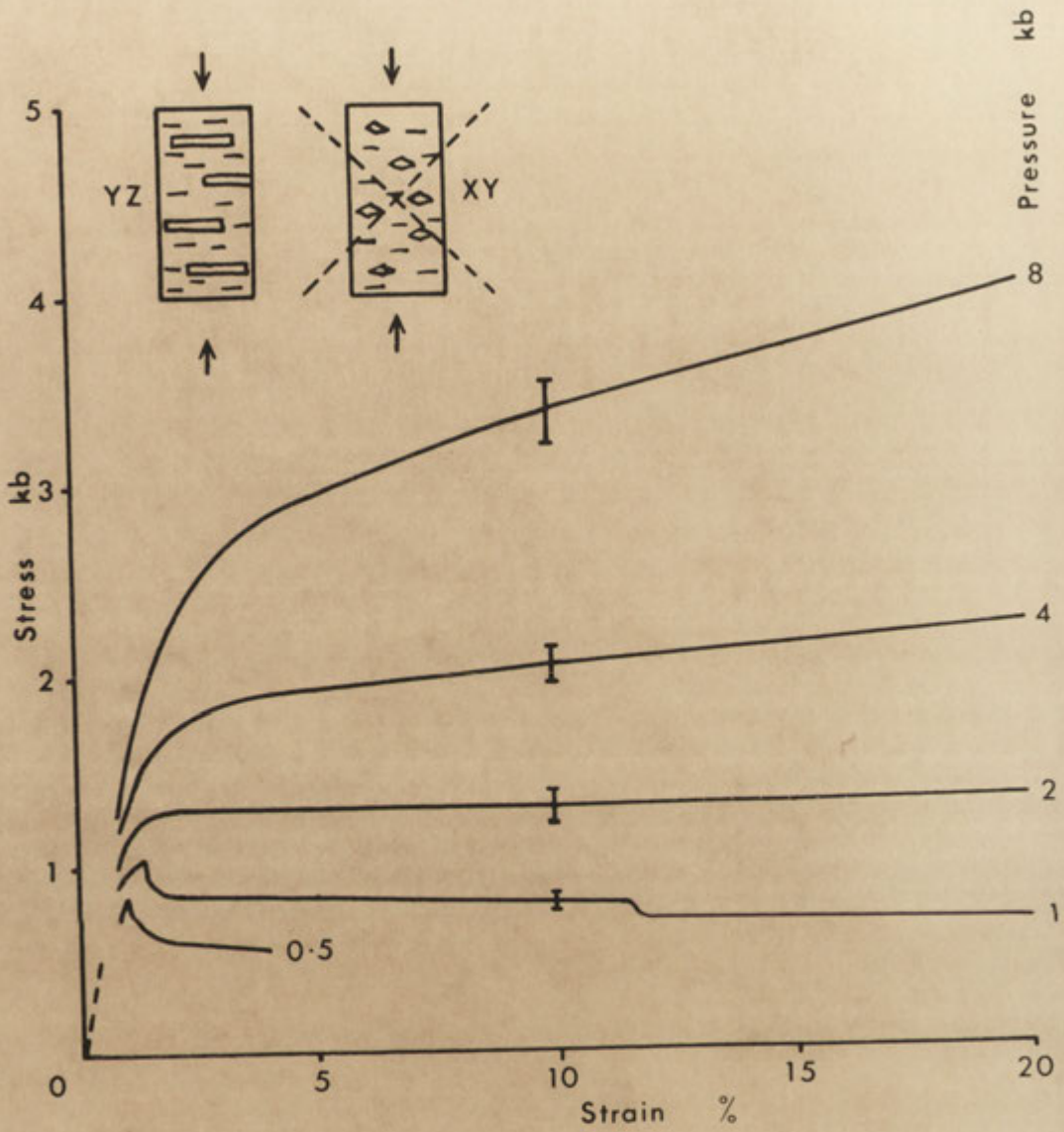


Fig. VI-3 Stress-strain curves for Y orientation.

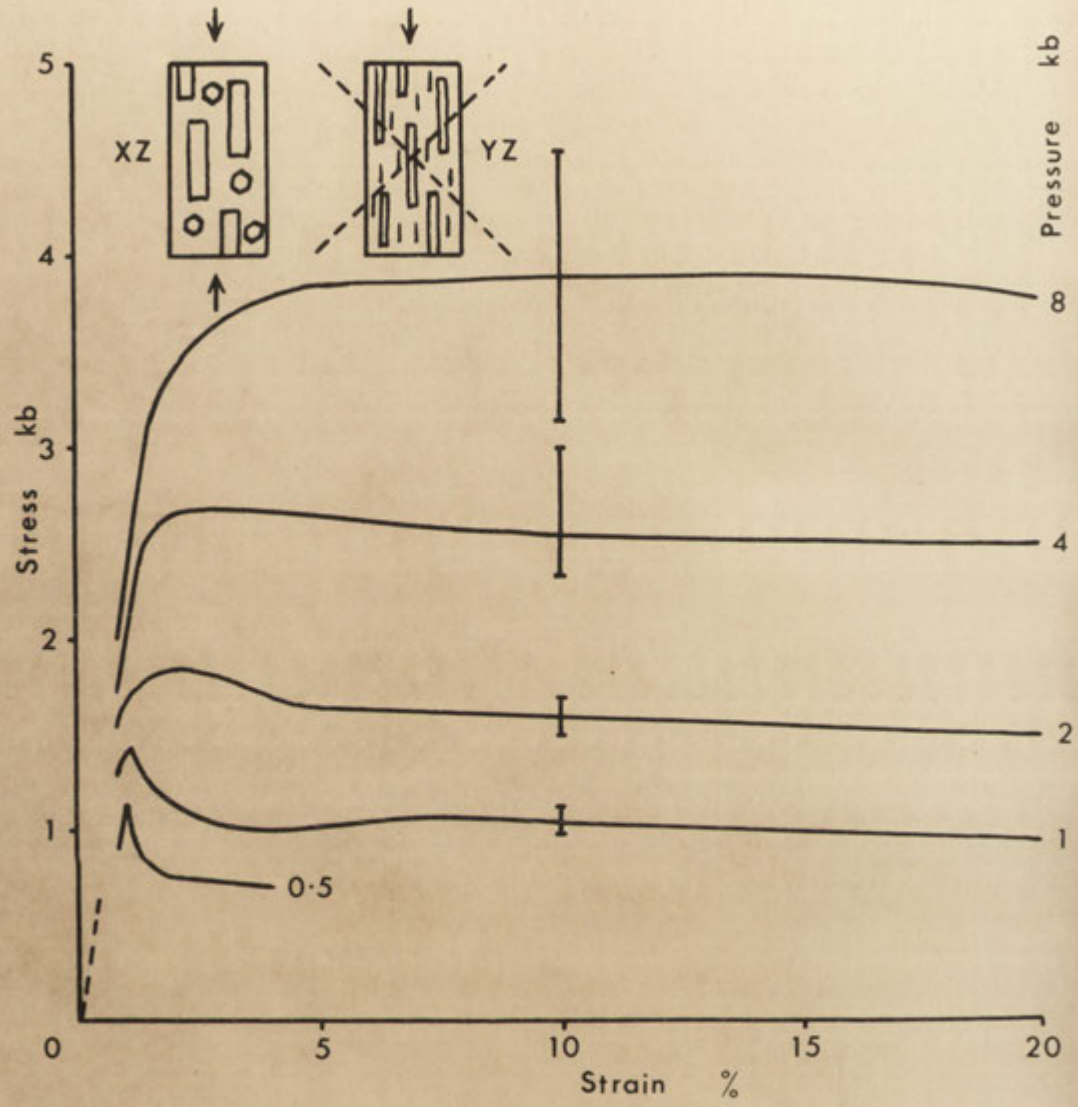


Fig. VI-4 Stress-strain curves for Z orientation.

CHAPTER VII

DEFORMATION
OF
OTHER PLATY MATERIALS

Introduction

These experiments were performed mainly because of the use of pyrophyllite, boron nitride and graphite in solid medium pressure apparatuses (see Chapter IX) and the general lack of published information on their stress-strain properties at high confining pressures. The results are of more general interest, however, in the context of the deformation of materials which are believed to have a number of independent slip systems far short of the five required to satisfy the von Mises' criterion.

Some of the pyrophyllite experiments reported in this chapter were performed by Dr. R. Coe.

Materials

Pyrophyllite Supplied by the American Lava Corporation as Grade A Lava (unfired). The bulk density was 2.69 g. cm^{-3} giving a theoretical porosity of about 5.3%. No preferred orientation of the pyrophyllite crystals was apparent in thin section and no anisotropy was detected on the stress-strain curves by deforming three specimens, cored in orthogonal directions, at a confining pressure of 4 kb. Specimens were made by grinding 10 mm diameter diamond drill cores to lengths of 20 mm. Specimens were air dried at room temperature for several weeks before use.

Boron nitride Supplied by Union Carbide (Australia). The

bulk density was 2.07 g. cm^{-3} giving a theoretical porosity of about 8.8%. Specimens were machined to 10 mm diameter and 20 mm long, all in the same direction relative to the original block. X-ray texture measurements showed a strong preferred orientation, twice as many crystals having their basal planes parallel to the specimen axis as perpendicular to it.

Graphite Supplied as ½ inch diameter rods by Morganite Carbon (Australia) as Electrographite Grade EY9. The bulk density was 1.73 g. cm^{-3} giving a theoretical porosity of about 23%. Specimens were machined (coaxially with the rod) to 10 mm diameter and 20 mm long. X-ray texture measurements showed that the graphite crystals had a strong preferred orientation, three times as many having their basal planes parallel to the specimen axis as perpendicular to it.

Techniques

Pyrophyllite Room temperature non volume change compression experiments as described in Chapter II. Three specimens were deformed at each of the confining pressures 0.5 kb, 1 kb, 2 kb, 4 kb and 8 kb. Red rubber jackets with a wall thickness of 1.6 mm were used at all confining pressures.

Boron nitride Room temperature non volume change

compression experiments as described in Chapter II. Two specimens were deformed at each of the confining pressures 2 kb, 4 kb, 6 kb and 8 kb. Annealed copper jackets with a wall thickness of 0.25 mm were used at all confining pressures.

Graphite Room temperature volume change compression experiments as described in Chapter II. Three specimens were deformed at each of the confining pressures 1 kb, 2 kb, 4 kb and 8 kb. Two supplementary room temperature non volume change compression experiments were also done as described in Chapter II using annealed copper jackets with a wall thickness of 0.25 mm i.e. with the same type of jackets as used in the volume change graphite experiments. One specimen was deformed at a confining pressure of 0.5 kb until failure occurred and one specimen was deformed at 4 kb until a downturn occurred on the load-displacement record.

Results

Pyrophyllite The means of the stress-strain curves at each confining pressure are drawn in Fig. VII-1; the vertical marker indicates the scatter which was about the same at all confining pressures. Specimens deformed at a confining pressure of 0.5 kb failed on a single slickensided shear plane inclined at about 40° to the

differential stress direction, with an associated stress drop on the stress-strain record. At 1 kb specimens barrelled slightly before failing on a single slickensided shear plane inclined at about 40° to the differential stress direction, the associated stress drop being less sharp than at 0.5 kb. At 2 kb specimens barrelled but showed traces of broad conjugate shear zones. At 4 kb and 8 kb the specimens barrelled and had continually rising stress-strain curves. The sensitivity of the flow stress to changes in the confining pressure was greater at larger strains but decreased with increasing confining pressure suggesting that cataclasis became of decreasing importance as the confining pressure was increased. The value of the pressure sensitivity ($\tan \psi$) at a confining pressure of 8 kb was 0.21 which is high compared with the value of 0.08 for talc under similar conditions. The reason for this large difference is not known.

Boron nitride All specimens deformed uniformly. The means of the pairs of stress-strain curves drawn in Fig. VII-2 rise continually and show no obvious elastic region or yield point. There appears to be a slight reduction in the sensitivity of the flow stress to changes in the confining pressure at higher confining pressures, but in view of the typical scatter of the original pairs

of stress-strain curves, as indicated by the vertical marker, this may not be a real effect.

Graphite The means of the stress-strain curves at each confining pressure are drawn in Fig. VII-3; the vertical marker indicates the scatter which was about the same at all confining pressures. Like the boron nitride, these curves rise continually and show no obvious elastic region or yield point. The one specimen deformed at a confining pressure of 0.5 kb failed on a single slickensided shear plane inclined at about 30° to the differential stress direction with an associated stress drop at about 4% strain. The sensitivity of the flow stress to changes of the confining pressure was slightly greater at the larger strains and initially decreased with increasing confining pressure but appeared to be fairly constant at the higher confining pressures.

After 20% deformation at a confining pressure of 1 kb the specimens were found to have sustained a permanent shortening, or set, of about 3%. At higher confining pressures a permanent set of about 1% was sustained after 20% deformation and to the naked eye the specimens showed no signs of having been deformed. The one specimen which was deformed 30% in compression at a confining pressure of 4 kb had a continually falling stress-strain curve after

about 24% strain, and a permanent set of about 7% remained associated with an overall volume increase of about 10%.

Excluding the two supplementary non volume change experiments, all specimens during deformation continually decreased in volume but at a decreasing rate (Fig. VII-4), i.e. they all have a negative value for $d^2v/d\varepsilon_A^2$ which leads to a strain hardening effect. The amount of the volume decreases decreased with increasing confining pressure. This is the reverse of the effect observed with materials in previous chapters. As the confining pressure was increased some of the initial porosity was eliminated leaving less porosity available for elimination by the differential stress at the higher confining pressures (Fig. VII-5). The structure of the material may be such that only part of this remaining porosity can easily be eliminated by stresses in the axial direction.

The total volume increase during reduction of the confining pressure tended to increase with the confining pressure at which the specimen had been deformed (see also frontispiece). In all cases most of the volume increase took place between 0.5 kb and atmospheric pressure.

Discussion

Like talc, all these materials are thought to have only one slip plane. In view of its physical resemblance

to talc it is noteworthy that the stress-strain properties of pyrophyllite at high confining pressures are quite dissimilar to those of talc. The barrelled appearance of the pyrophyllite after deformation at a confining pressure of 8 kb constrasts with the more uniform distribution of the deformation in the talc specimens after deformation at confining pressures of 6 kb and above. Some correlation seems to exist between uniformly distributed deformation and volume decreases during these experiments: if an extrapolation is made to the case of pyrophyllite the shape of the deformed specimens would be indicative of volume increases and therefore cataclastic deformation. This tentative conclusion is supported by the relatively high pressure sensitivity of pyrophyllite's flow stress.

Graphite is known to possess the family of slip systems $\{0001\}\langle 11\bar{2}0\rangle$ (Groves and Kelly, 1963, in reference to an unpublished work by Freise) which gives three physically different slip systems, all with slip directions lying parallel to the basal planes, only two of which are independent. Boron nitride, which is also a hexagonal material may be expected to possess a similar family of slip systems in view of its obvious basal glide plane. With both of these materials, and particularly in the case of graphite, the preferred orientation of the crystals

is favourable for deformation by kinking and bending of the crystals about axes lying parallel to the basal planes. In the case of the graphite, however, the almost complete recovery of the specimens back to their pre-20% deformation length suggests that kinking could not have been the primary mechanism. It is possible that deformations of 20% in the graphite could take place using what is essentially an elastic mechanism. Most of the crystals were being loaded at an acute angle to their basal planes and considerable shortening parallel to these planes could be obtained by flexure of the crystals with some slip on the basal planes. The problem of compatibility would be alleviated to some extent by the presence of substantial porosity even at a confining pressure of 8 kb. After deformation the new configuration of the crystals, which have low flexural stiffness about all axes lying in the basal planes, would be effectively "frozen in" and unflexing occurs only when friction is reduced by lowering the confining pressure. It appears from the specimen which was deformed 30% at a confining pressure of 4 kb that irreversible deformation commenced after about 24% strain.

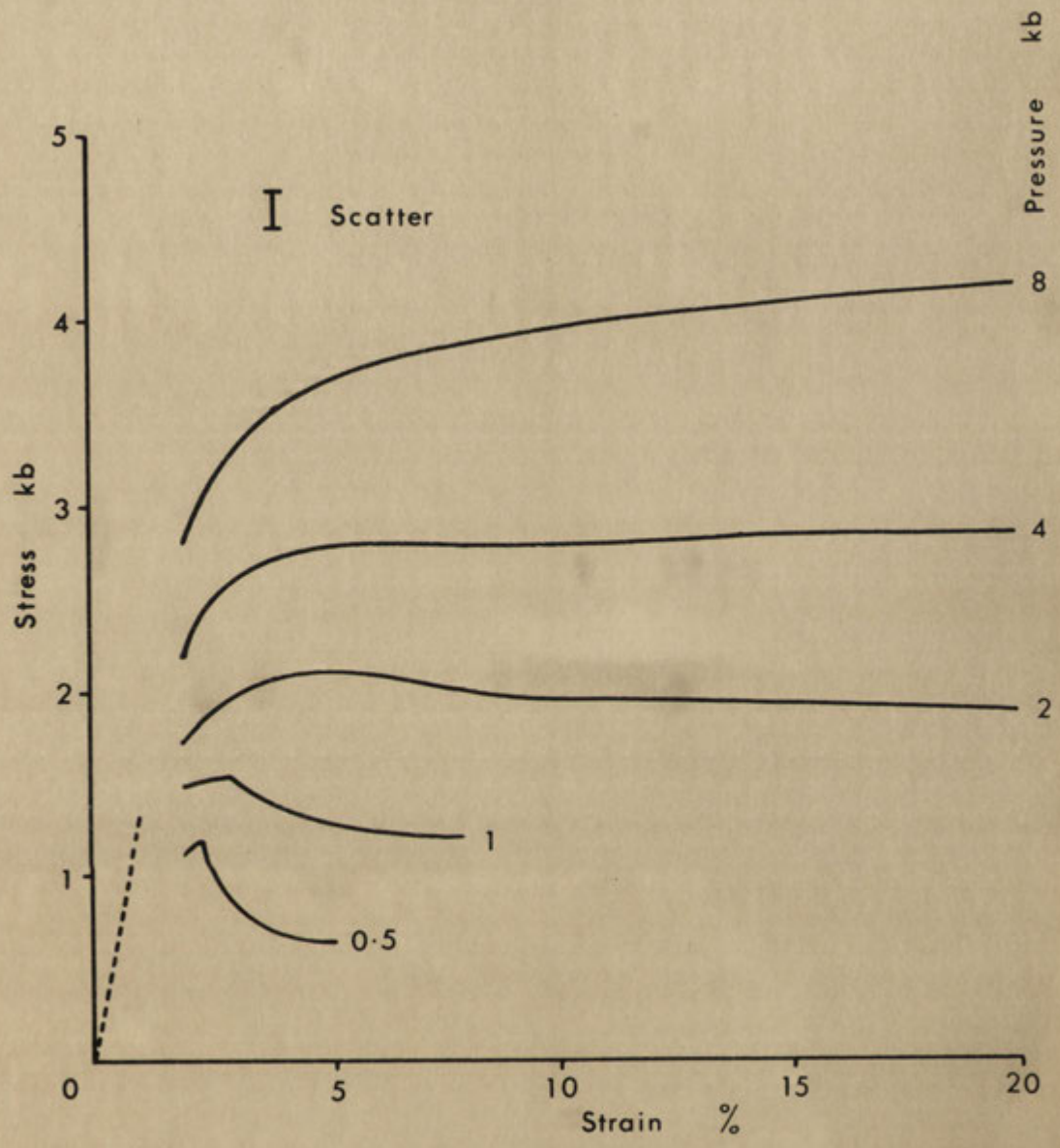


Fig. VII-1 Stress-strain curves for pyrophyllite.

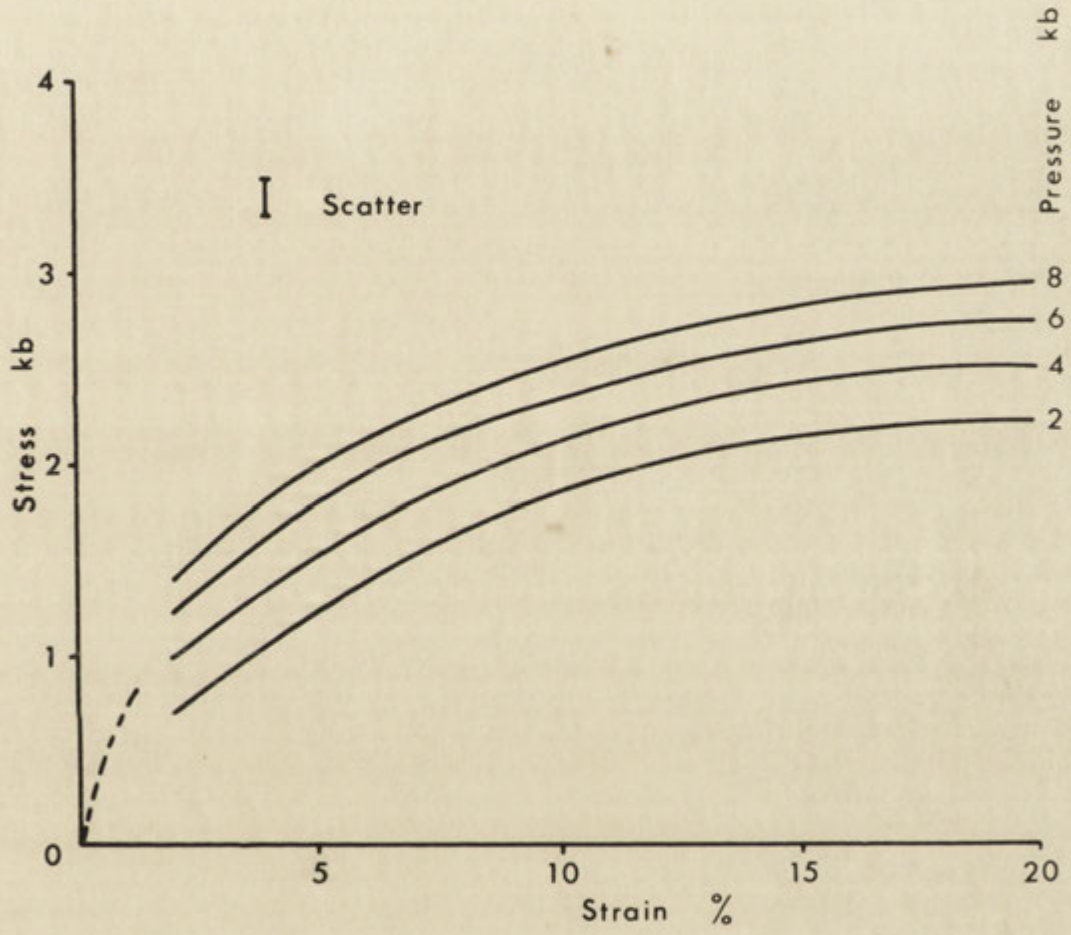


Fig. VII-2 Stress-strain curves for boron nitride.

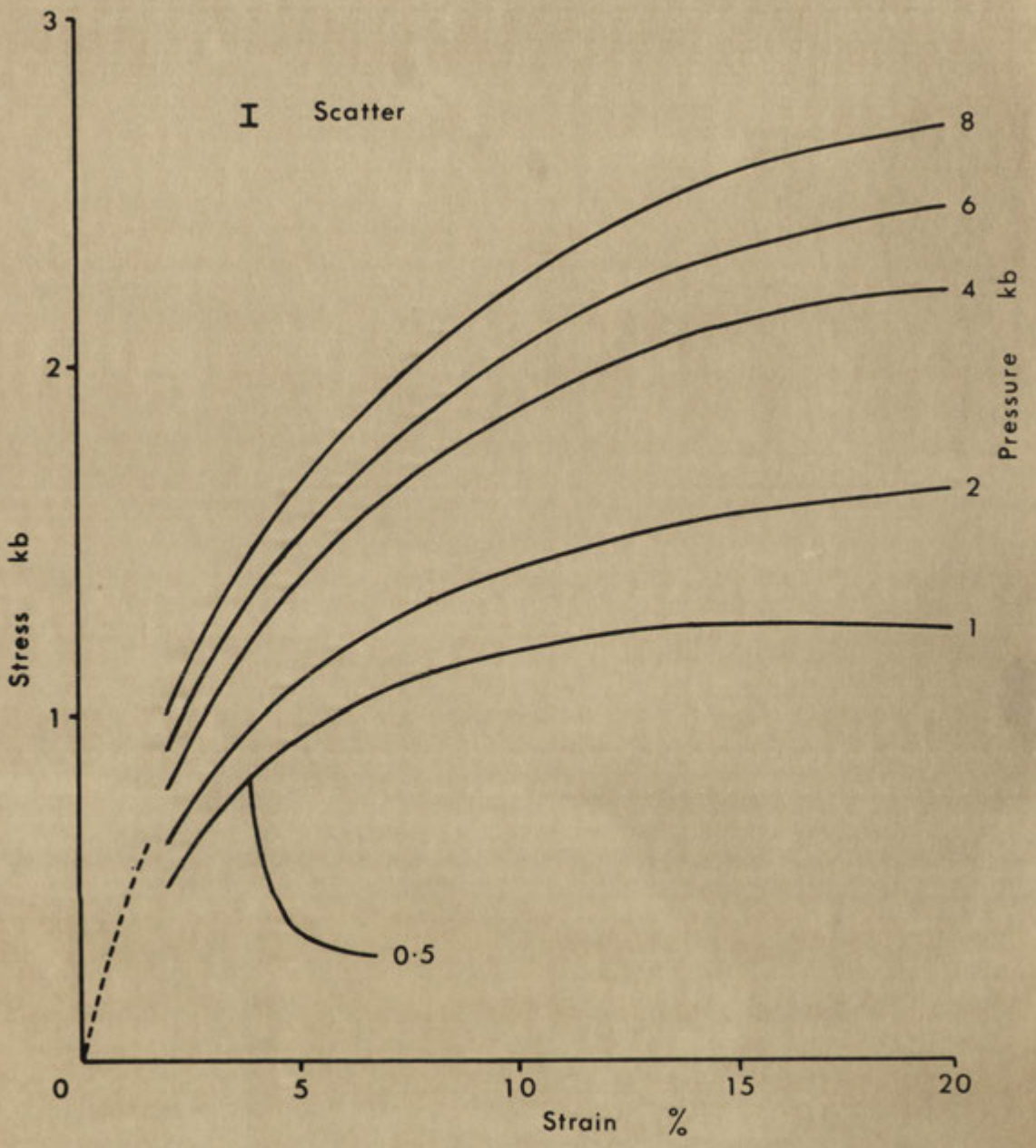


Fig. VII-3 Stress-strain curves for graphite.

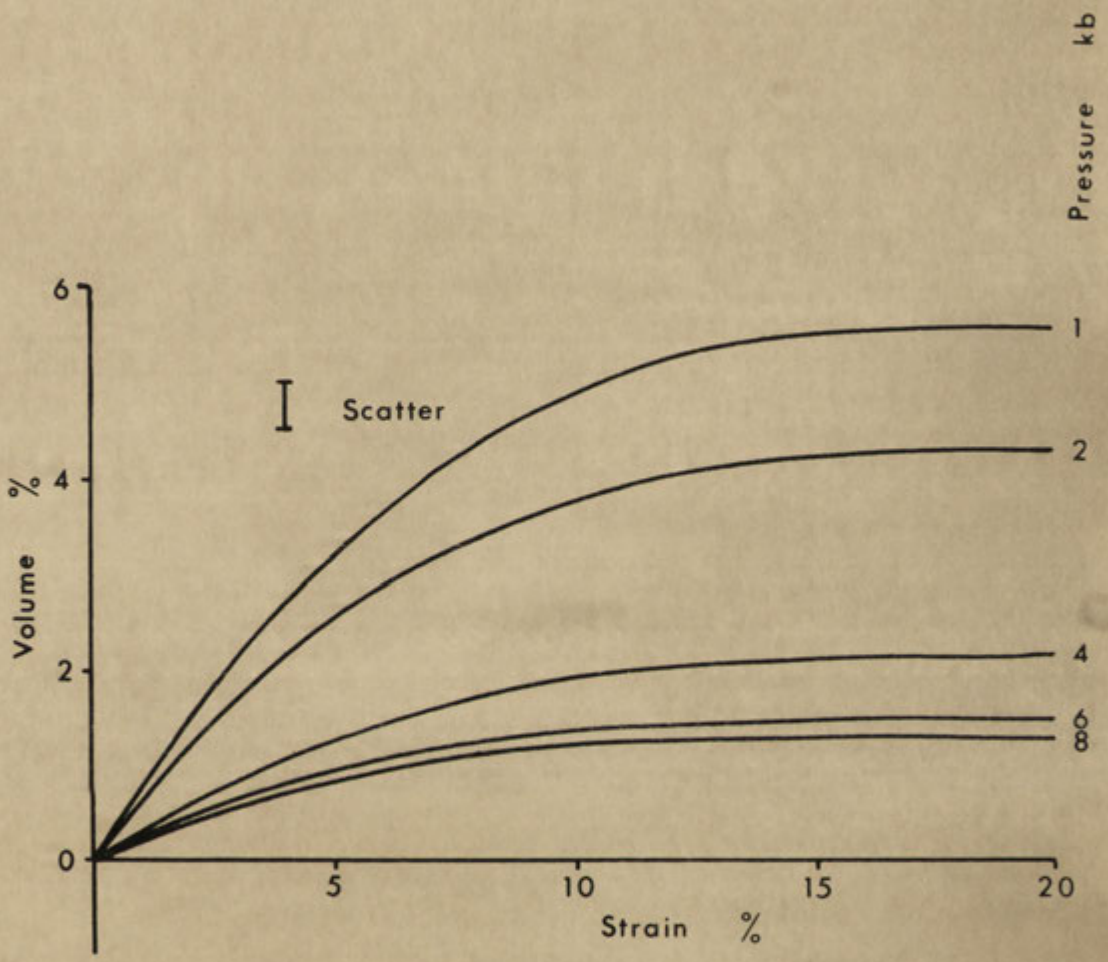


Fig. VII-4 Volume change-strain curves for graphite.

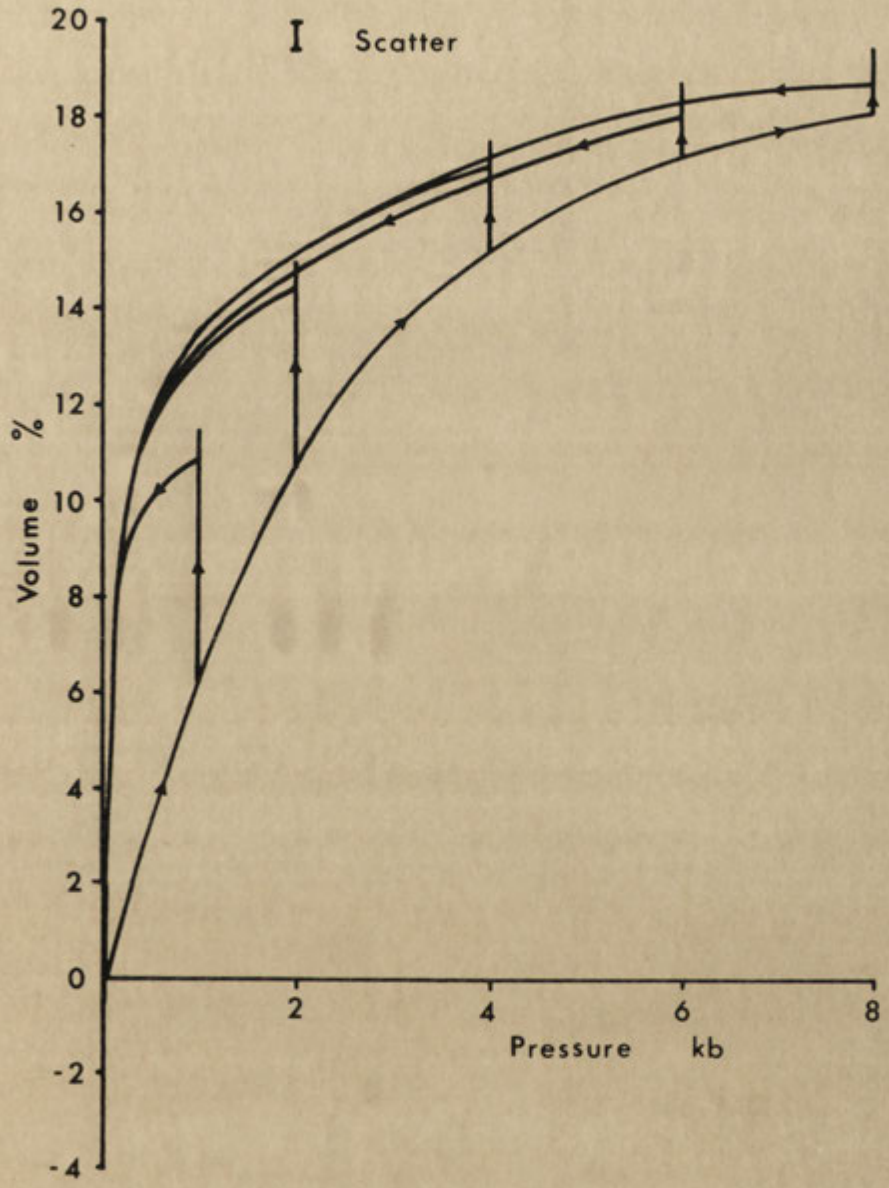


Fig. VII-5 Overall volume changes for graphite.

CHAPTER VIII

DEFORMATION
OF
SODIUM CHLORIDE AND SILVER CHLORIDE

Introduction

These experiments were done mainly because of the use of sodium chloride and silver chloride in solid medium pressure apparatuses (see Chapter IX). Their isometric structure, and well documented slip systems, however, make comparison of these results with those from other materials with lower symmetry of some interest.

Materials

Sodium chloride Analar analytical reagent which had been dried for one hour at 110°C and then stored in a desiccator was used. The work of Ammar and Budworth (1967) indicated that pressure sintering at room temperature was a suitable process for making specimens with close to theoretical density. The specimens used for these experiments were made by weighing out quantities of sodium chloride required to make specimens 10 cm diameter and 20 mm long with 100% theoretical density. This was placed inside a specially designed piston-cylinder press having an inside diameter of 10 mm, (Fig. VIII-1). The pressing process was standardised as far as possible in order to produce as nearly identical specimens as possible. After evacuating the cylinder, a nominal pressure of 10 kb was applied and held constant for 20 minutes before being slowly reduced. The inside wall of the cylinder had been ground to a mirror finish and was

lubricated with a thin film of molybdenum disulphide compound, but a nominal pressure of about 2 kb was required to push the pistons and the specimen down out of the cylinder. Some specimens were found to be cracked or broken on planes perpendicular to the cylinder axis and were rejected. The ends of the remainder were lightly ground to remove the impressions made by the piston mitre rings. The bulk density of these specimens was 2.15 g. cm^{-3}, giving a theoretical porosity of about 0.6%. This porosity is probably partly due to small scale cracking of the specimen during removal from the piston cylinder press. The specimens were not annealed before use.

Silver chloride A commercially pure grade supplied by Townson and Mercer Pty. Ltd. (Australia) was used. Specimens were made by melting the silver chloride, pouring it into a mould and finally machining it to 10 mm diameter and 20 mm long cylinders. The process was standardised as far as possible in order to produce as nearly identical specimens as possible. This was the method used to make silver chloride pressure cells for the solid medium apparatus in this department. The bulk density of these specimens was 5.41 g. cm^{-3}, giving a theoretical porosity of about 2.7%.

Techniques

Sodium chloride Room temperature volume change compression

experiments and jacketing of specimens as described in Chapter II. Two specimens were deformed at each of the confining pressures 0.25 kb, 0.5 kb, 1 kb, 2 kb, 6 kb and 8 kb.

Silver chloride Room temperature non volume change compression experiments as described in Chapter II. Two specimens were deformed at each of the confining pressures 0.5 kb, 1 kb, 2 kb, 4 kb and 8 kb. Annealed copper jackets with a wall thickness of 0.25 mm were used at all confining pressures.

Results

Sodium chloride All the specimens deformed uniformly with continually rising stress-strain curves (Fig. VIII-2). Some scatter, as indicated by the vertical marker, occurred which obscured any dependence that the curves may have on the confining pressure. It is, however, possible to place an upper limit of about 0.01 on the pressure sensitivity tan ψ.

All specimens decreased in volume at a decreasing rate during deformation (Fig. VIII-3). Because of the scatter no clear difference could be detected between volume changes taking place during deformation at different confining pressures, but there was some tendency for the volume decreases to be smaller at the higher confining pressures.

The overall volume change curves (Fig. VIII-4) are distinctly different from those for all the other materials tested. During increase of the confining pressure some

porosity was eliminated. The remainder was eliminated during deformation. When the confining pressure was reduced the increase in volume which occurred was only due to elastic effects (see also frontispiece). The final porosity of the specimens, obtained from density measurements, was in every case less than 0.1%. The volume changes due to elastic effects shown in Figs. VIII-3, 4 were calculated using the Hashin bounds for sodium chloride (Simmons, 1967), assuming that the specimens were isotropic.

Silver chloride All the specimens deformed uniformly with continually rising stress-strain curves. The means of the pairs of stress-strain curves at each confining pressure are drawn in Fig. VIII-5, the vertical marker indicating the scatter. Increasing the confining pressure caused a small but measurable increase in the flow stress particularly at small strains. This was probably partly due to elimination of porosity during the early stages of deformation. During the later stages of deformation the pressure sensitivity was about 0.01.

Discussion

Polycrystalline sodium chloride is brittle at atmospheric pressure and room temperature, but becomes ductile when subjected to relatively low confining pressures. Schmidt (1937) and Goguel (1948) did triaxial compression experiments

using polycrystalline sodium chloride (the specimens used by Schmidt had been annealed), but their experiments were not continued beyond about 4% strain. Handin (1953) using a natural rock salt obtained very similar stress-strain curves to those in Fig. VIII-2.

Polycrystalline silver chloride is brittle at temperatures below about -100°C at atmospheric pressure (Stokes and Li, 1963) but ductile at room temperature and atmospheric pressure.

The stress-strain curves for both the sodium chloride and silver chloride have very low pressure sensitivities which suggests that intra crystalline plasticity is the mechanism of deformation. In the case of the sodium chloride this has been confirmed by volume change measurements, the specimens continuing to deform at zero porosity with no tendency for the volume to increase.

Gilman (1961) published a review of the mechanical behaviour of ionic crystals, and several workers, including more recently Handin and Hager (1958) and Davis and Gordon (1968) have demonstrated that the $\{110\}\langle 1\bar{1}0\rangle$ family of slip systems in sodium chloride is easily activated at atmospheric pressure and room temperature and has a very low pressure sensitivity. This family of slip systems has six physically distinct slip systems but only two of them

are independent (Groves and Kelly, 1963). This family of slip systems therefore falls a long way short of providing the five independent slip systems required by the von Mises' criterion and cataclasis becomes involved during deformation at atmospheric pressure and room temperature. Sodium chloride also possesses the $\{001\}\langle 1\bar{1}0\rangle$ family of slip systems which is not normally active at atmospheric pressure and room temperature. As the confining pressure is increased, however, the stresses required to cause cataclastic deformation increase relatively rapidly and the $\{001\}\langle 1\bar{1}0\rangle$ family of slip systems becomes active. For example, Scott and Pask (1963) and Paterson and Weaver (1969) have demonstrated that this family of slip systems can be activated in the structurally similar lithium fluoride and magnesium oxide respectively, when they are subjected to severe stress concentrations at room temperature. This family of slip systems is also known to be active at atmospheric pressure and room temperature in silver chloride (eg. Stokes and Li, 1963). In conjunction with the $\{110\}\langle 1\bar{1}0\rangle$ family of slip systems this satisfies the von Mises' criterion.

We therefore deduce that the $\{001\}\langle 1\bar{1}0\rangle$ family of slip systems is activated in some of the crystals in our work hardened polycrystalline sodium chloride when it is

subjected to confining pressures of 0.25 kb (the lowest confining pressure used in these experiments). The confining pressure required to activate this family of slip systems in annealed polycrystalline sodium chloride specimens would not be expected to be higher than that required for the work hardened material because annealing should make the initial activation of a slip system easier without affecting the pressure sensitivity of the stresses required for cataclastic deformation.

The absence of any non-elastic volume increases during decrease of the confining pressure after deformation is consistent with the isotropy of linear compressibility of the sodium chloride crystals. Thus, when the confining pressure is changed each crystal will change its dimensions equally in all directions and no further intergranular stresses will arise.

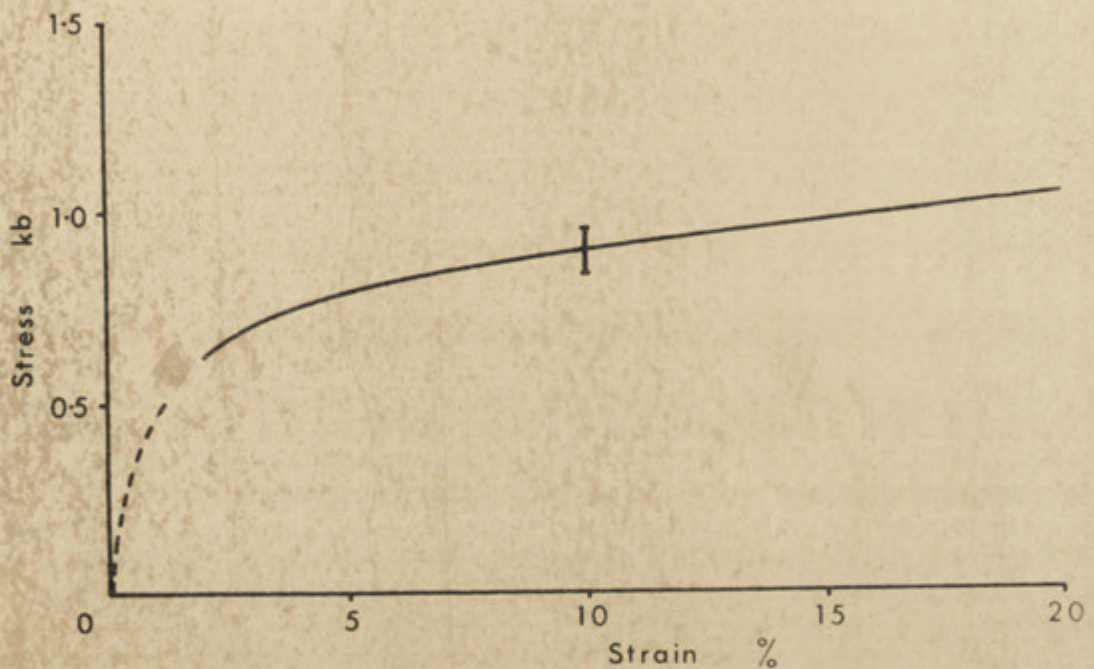


Fig. VIII-2 Stress-strain curves for sodium chloride.

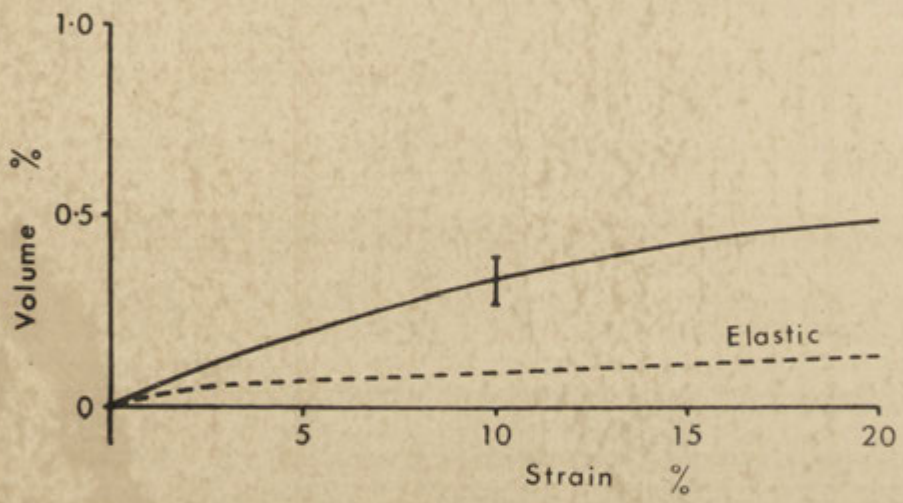


Fig. VIII-3 Volume change-strain curves for sodium chloride.

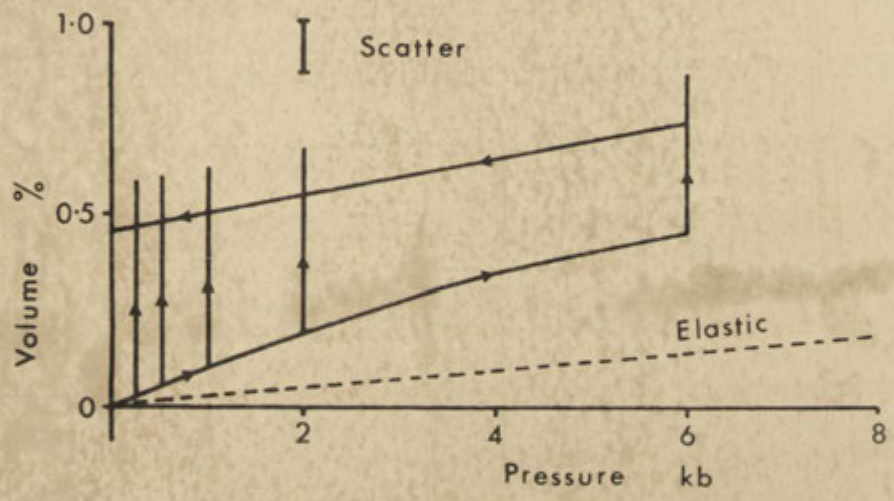


Fig. VIII-4 Overall volume changes for sodium chloride.

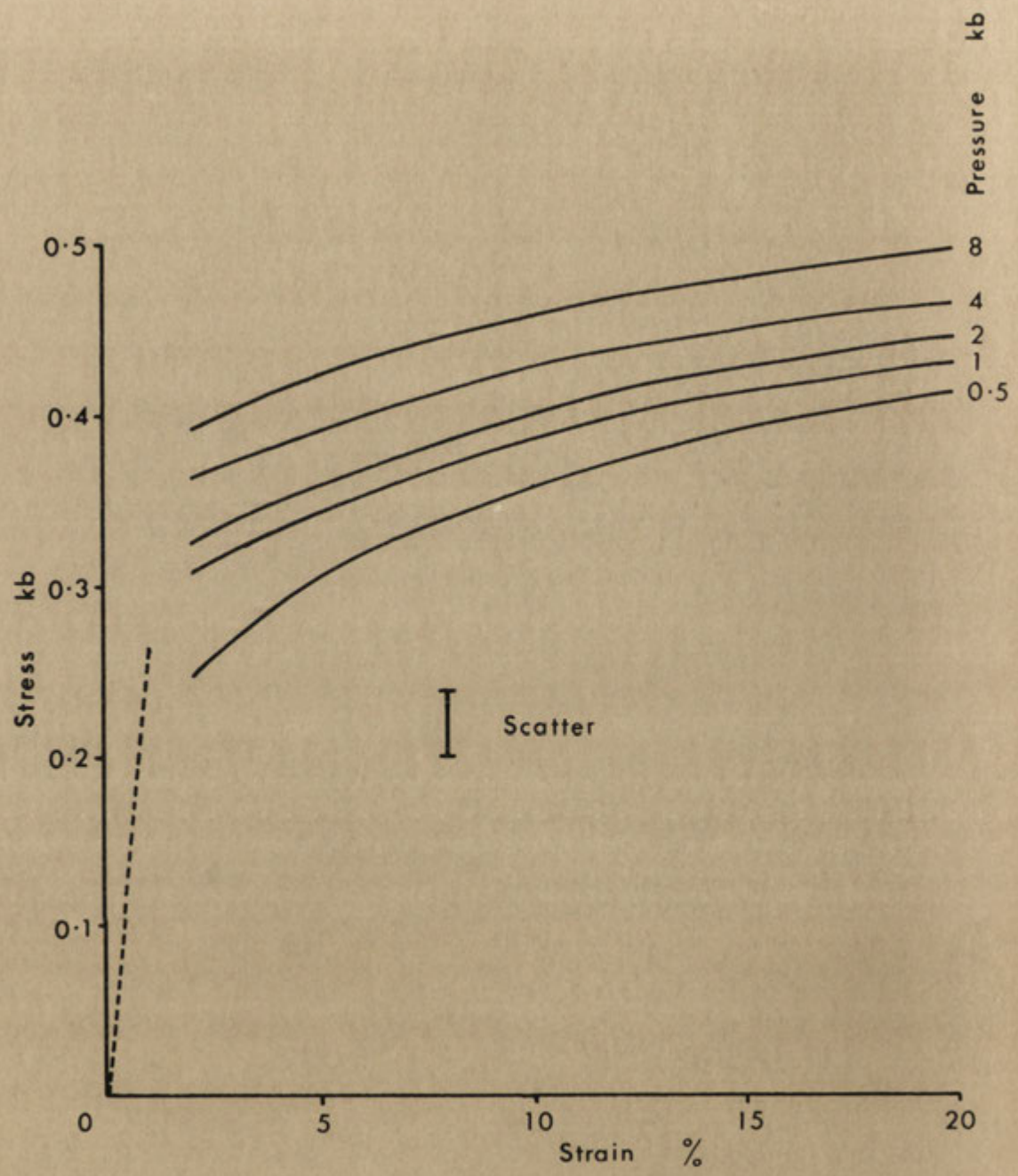


Fig. VIII-5 Stress-strain curves for silver chloride.

CHAPTER IX

APPLICATION

TO

SOLID PRESSURE MEDIA EXPERIMENTS

Solid media are widely used in experiments at various temperatures at high pressures and especially above 10 kb. The materials used are chosen for their alleged low or negligible strength. There is, however, little published information on the stress-strain properties of these materials under pressure although measurements on shearing discs give some indications. Bridgman (1935, 1937) did some tests on silver chloride, sodium chloride, graphite and lead; and Vereshchagin and Zubova (1960-1961) did some tests on silver chloride, pyrophyllite and graphite. Graf and Hulse (1964, 1965) tested pyrophyllite and talc in compression at atmospheric pressure at a variety of temperatures, but since they ignored the effects of confining pressure and of vapour pressure resulting from decomposition at high temperatures their results are of doubtful relevance.

Recent controversy about the magnitude of corrections which should be made to the nominal pressures to compensate for the strength of the solid media emphasised the need for further information. Most workers using piston cylinder solid medium pressure apparatuses of the Boyd and England type (1960a) calculate the nominal pressure in the solid medium by dividing the force applied to the piston by its cross sectional area and then make various empirical corrections for friction between the piston and cylinder, for

pressure losses due to friction within the solid medium, and for friction between the solid medium and the cylinder walls (Tamayama and Eyring, 1967). Some controversy exists concerning these corrections. Green, Ringwood and Major (1966) quote a friction correction of -10% of the nominal pressure at 35 kb and 1100°C, but state that this correction should be essentially independent of temperature; Klement, Jayaraman and Kennedy (1963) quote a friction correction which decreases from -12% of the nominal pressure at 17 kb to about -7% at 41 kb, no variation with temperature being recorded; Kitahara and Kennedy (1964) quote a friction correction of - 9½% of the nominal pressure at 500°C and -7½% at 1100°C; Pistorius (1967) quotes a friction correction of -0.8 kb to -2 kb which is independent of the nominal pressure, no variation with temperature being recorded; Boettcher and Wyllie (1968) quote friction corrections of -13% of the nominal pressure at room temperature and -7% at 850°C; Newton (1965) in work at pressures of 4 kb to 8 kb and temperatures of 640°C to 860°C applied a pressure correction of -1.5 kb representing a -37% to -19% correction; Newton and Smith (1967) quote a friction correction of -10% of the nominal pressure up to 30 kb which is independent of temperature; Boyd and England (1960a, 1960b) quote friction corrections of -13%

of the nominal pressure at room temperature and -8% at higher temperatures, but in a later work (1963) they concluded that no correction was required at high temperature.

Experiments recorded in this thesis on talc, pyrophyllite, boron nitride, silver chloride, sodium chloride and graphite do not represent the normal conditions under which these materials are used in solid medium pressure apparatuses, but it is considered that the extrapolations which can be made from these experiments permit more realistic estimates than hitherto of their properties at higher pressures.

The reduction in the pressure sensitivity of some of these materials with increasing confining pressure makes accurate extrapolation difficult in some cases since the strength will not increase linearly with confining pressure. It is, however, possible to put lower and upper limits on the room temperature strength for a given confining pressure with reasonable confidence. If it is assumed that the material under consideration will not be weaker at the extrapolated pressure than it is at the maximum confining pressure in our experiments this provides a lower limit. If it is assumed that the pressure sensitivity, tan ψ, will not increase above its value at the maximum confining pressure in our experiments, a linear extrapolation to the higher pressure based on this pressure sensitivity provides

an upper limit. Fig. IX-1 gives the lower and upper limits at an extrapolated pressure of 25 kb. Although many experiments in solid medium pressure apparatuses are performed at high temperatures, these temperatures are localised near the furnace and are not representative of the temperature in most of the solid medium.

The capacity of the solid medium to support a significant shear stress has several consequences. Deviations occur from the nominal hydrostatic pressure due to a number of factors:-

1. Friction between the piston and cylinder (this is independent of the properties of the solid medium and will not be considered in detail).

2. Friction within the solid medium and the behaviour of the specimen.

3. Friction between the solid medium and the inside wall of the pressure vessel.

4. The strength of the graphite furnace

The final friction correction will also be affected by time dependent effects such as creep, and the experimental technique used.

1. Friction between the piston and cylinder

This will depend to a large extent on the design of the apparatus and the type of seal used. Friction corrections

at moving piston seals quoted by Paterson (1962, 1964a) would suggest a partial friction correction, F_I, of about -1.5% of the nominal pressure.

2. Friction within the solid medium and the behaviour of the specimen

This is highly complex and it is difficult to make anything but broad generalisations.

The pressure losses arising within the solid medium will depend basically on the flow patterns within the solid medium. These will be affected by the properties and behaviour of all the internal components of the pressure cell including the specimen itself and will also depend on the experimental technique used. Some distortion of the original configuration of the internal components is to be expected due to differences in initial porosities and the different flow stresses of various components.

If the specimen assembly is strong compared with the surrounding solid medium and if it is behaving isotropically, then the axial stress (with respect to the cylinder axis) may exceed the radial stress during piston advance by as much as the flow stress of the surrounding solid medium under the prevailing conditions of pressure and temperature. Clearly it is advantageous to use the weakest suitable solid medium. From Fig. IX-1 the flow stresses at 25 kb of the

solid media tested and their order of preference solely on the basis of their flow stresses at room temperature would be as follows:-

1.	Silver chloride	0.5 kb
2.	Sodium chloride	1.0 kb
3.	Graphite	2.8 kb
4.	Talc	3.0 kb
5.	Boron nitride	3.4 kb
6.	Pyrophyllite	5.2 kb

In practice talc is most commonly used as the actual pressure medium. The decomposition of talc to give quartz, enstatite and water, which occurs at temperatures increasing from approximately 750°C to 850°C as the confining pressure increases to 30 kb, further complicates the situation. Fig. V-1 shows that the flow stress of talc at 4 kb confining pressure decreases steadily to about one third of its value at room temperature as the temperature is increased to about 600°C. At temperatures from 600°C to 900°C the flow stress appears to be fairly constant. The flow stress is not affected substantially by venting the specimens. It must be emphasised that the high temperature experiments with talc described in chapter V may not be representative of conditions at higher confining pressures. We will assume, however, that changes similar to those in Fig. V-1 take

place in the flow stress of talc as the temperature increases at higher confining pressures. Because of the large temperature gradients only talc in the immediate vicinity of the furnace is likely to be decomposed even during experiments at very high temperatures. If we assume therefore that the talc will retain at least one third of its room temperature flow stress, at 25 kb it would be at least 1 kb. The specimen could therefore be subjected to a differential stress of at least 1 kb during the final stages of piston advance to 25 kb and serious deformation of the specimen could occur. This differential stress would be nearer 2 kb or 3 kb at lower temperatures.

If the flow stress of the solid medium is σ_0 under the prevailing conditions of pressure and temperature the axial stress and radial stresses will be σ_N and $(\sigma_N - \sigma_0)$ respectively, where σ_N is the nominal pressure if we assume that there are no other frictional losses.

The mean stress on the specimen will therefore be

$$1/3\{\sigma_N + 2(\sigma_N - \sigma_0)\} = \sigma_N - 0.67\sigma_0$$

and the partial friction correction F_{II} can therefore be written

$$F_{II} = - \frac{0.67\sigma_0}{\sigma_N} \times 100\%$$

This correction is rather insensitive to variations in nominal pressure and will therefore be proportionally larger at lower pressures. It will, however, vary to some extent with temperature.

Taking values:-

σ_N = 25 kb

σ_0 = 1 kb (talc during an experiment at high temperature)

F_{II} = - 2.7%

If the specimen is weaker than the solid medium the maximum differential stress it can be subjected to is of course limited by the flow stress of the specimen rather than that of the solid medium. Any anisotropic shape changes or volume changes of the specimen could radically alter the state of stress in the solid medium. For example a radial expansion of the specimen such as that occurring during deformation in a Griggs' (1967) type apparatus would induce an immediate increase in the radial confining stress.

3. Friction between the solid medium and the inside wall of the pressure vessel

A sheath of lead foil or molybdenum disulphide compound is sometimes used to minimise friction at this interface but more frequently no lubrication other than that due to the solid medium itself is present.

Considering the simplified pressure cell in Fig. IX-2:

Let the nominal pressure $\sigma_N = \frac{F}{\pi r^2}$

Let the quasi-hydrostatic stress at the specimen S situated distance x from the piston face be σ_s. If the flow stress of the solid medium at the interface, under the prevailing conditions of pressure and temperature, is σ_0', we assume that a limiting shear stress $0.5\sigma_0'$ is attained over the entire interface during the final increment of the nominal pressure. We further assume that there are no other frictional forces arising in this model.

Now:-

$$\sigma_N \pi r^2 = \pi r x \sigma_0' + \sigma_s \pi r^2$$

$$\sigma_s = \frac{\sigma_N r - x\sigma_0'}{r}$$

$$= \sigma_N - \frac{x\sigma_0'}{r}$$

The partial friction correction, F_{III}, arising from friction between the solid medium and the inside wall of the pressure vessel, assuming that no other corrections to the nominal pressure are made, can therefore be written:-

$$F_{III} = - \frac{x\sigma_0'}{r\sigma_N} \times 100\%$$

This correction is rather insensitive to variations in the nominal pressure and temperature, and will therefore be proportionally larger at lower nominal pressures, but it is very dependent on the geometry of the pressure cell, the correction being directly proportional to the x/r ratio. Newton (1966, p. 208) found a considerable reduction in his overall friction correction when he used a larger diameter pressure cell with the same solid medium.

Consider for example a pressure cell with dimension similar to that used by Green et al. (1966, p. 3590).

Taking values:-

$$\sigma_N = 25 \text{ kb}$$

$$\sigma_0' = 3 \text{ kb (talc)}$$

$$r = 0.635 \text{ cm}$$

$$x = 1.52 \text{ cm}$$

$$F_{III} = -2.9\%$$

During a decompression stroke the pressure in the solid medium is not necessarily relieved by shear at the interface at the inside wall of the cylinder. The alternative is for the body of the solid medium to flow and follow the piston face; this it will do if the stress exerted by the piston on the solid medium falls below the

radial stress still existing in the solid medium by an amount equal to the flow stress of the medium at that confining pressure. Under some circumstances this would be the easier mechanism and the friction correction for the decompression stroke would be less than for the compression stroke (i.e. not as generally assumed equal to it). It may thus be possible to avoid the worst effects of wall friction by using a "decompression method" i.e. applying a nominal pressure greater than that required and then withdrawing the piston slightly. This, and similar methods are described by Green et al. (1966).

4. The strength of the graphite furnace

It has been stated that the graphite furnace will have substantially less strength than the cooler talc (Green et al., 1966, p. 3593). In actual fact the strength of the graphite furnace is more likely to increase with temperature. Mantell (1958) states that at atmospheric pressure the strength of graphite reaches a maximum at 2500°C at which temperature it has approximately double its room temperature strength.

Carter, Christie and Griggs (1964) have noted extension fractures in quartz specimens after deformation in a solid medium apparatus which they attributed to stresses arising from expansion of the graphite furnace during pressure

release. This explanation is supported by observations in Chapter VII of unusually large strain recoveries in graphite during release of pressure.

Considering the simplified pressure cell in Fig. IX-3 at a nominal pressure of σ_N and assuming that no other corrections to the nominal pressure are made:-

Let the cross sectional area of the graphite = G

Let the flow stress of the graphite at σ_N = σ_0''

Differential load supported by graphite column = $\sigma_0'' G$

Over the area of the piston this represents a stress

$$= \frac{\sigma_0'' G}{\pi r^2}$$

The partial friction correction F_{IV} arising from this factor can therefore be written:-

$$F_{IV} = - \frac{\sigma_0'' G}{\pi r^2 \sigma_N} \times 100\%$$

This correction is rather insensitive to variations in the nominal pressure and will therefore be proportionally larger at lower nominal pressures.

Consider for example a pressure cell with dimensions similar to that used by Green et al. (1966).

Taking values:-

$$\sigma_N = 25 \text{ kb}$$

$\sigma_0'' = 2.7$ kb (room temperature value)

$r = 0.635$ cm

$G = 0.19$ cm^2

$$F_{IV} = -1.6\%$$

If we assume that these partial friction corrections are additive, we can now obtain a theoretical nominal pressure correction F

where $F = F_I + F_{II} + F_{III} + F_{IV}$

The examples we have calculated refer to an apparatus with dimensions similar to that used by Green et al. (1966) during piston advance at a nominal pressure of 25 kb and a temperature of about 1000°C using talc as the pressure medium.

$$F = -1.5\% - 2.7\% - 2.9\% - 1.6\%$$

i.e. $$F = -8.7\%$$

It is perhaps fortuitous that this correction corresponds so closely with the -10% obtained by Green, Ringwood and Major (1966) by empirical means in view of the simplifying assumptions made in the above calculations.

Conclusions

1. The strength of any solid medium material may be expected to vary according to its source, but it is unlikely that any material can be considered to have negligible strength under all circumstances.

2. The stress state at any point may depart from the hydrostatic state by as much as the flow stress of the medium at that confining pressure and temperature and will also depend on the experimental technique and behaviour of the specimen.

3. Friction corrections are not necessarily the same on the compression and decompression strokes. Friction during compression will usually be the larger.

4. The complete friction correction will consist mainly of an 'apparatus factor', plus a small proportion of the nominal pressure. Expressed as a percentage of the nominal pressure the complete friction correction will decrease with increasing nominal pressure. Some reduction should also result from increasing the temperature.

5. The complete friction correction may be reduced for a given apparatus and pressure cell by modifying the experimental technique.

6. Whilst it is not a conclusion, it should be noted that time dependent effects such as creep would be expected to ameliorate all non-homogeneous and non-hydrostatic stress states.

Material	Flow stress at 10% strain at 8 kb (at 10 kb for talc) (kb)	Tan ψ at 10% strain at 8 kb (at 10 kb for talc)	Extrapolation to give strength at 10% strain and 25 kb (kb)
Three Springs talc Block A	2.35	0.08	3.5
Pyrophyllite	3.95	0.21	7.5
Boron nitride	2.55	0.10	4.3
Silver chloride	0.47	0.01	0.6
Sodium chloride	0.90	<0.01	1.0
Graphite	2.24	0.06	3.3

Fig. IX-1. Summary of room temperature results with extrapolation to 25 kb confining pressure.

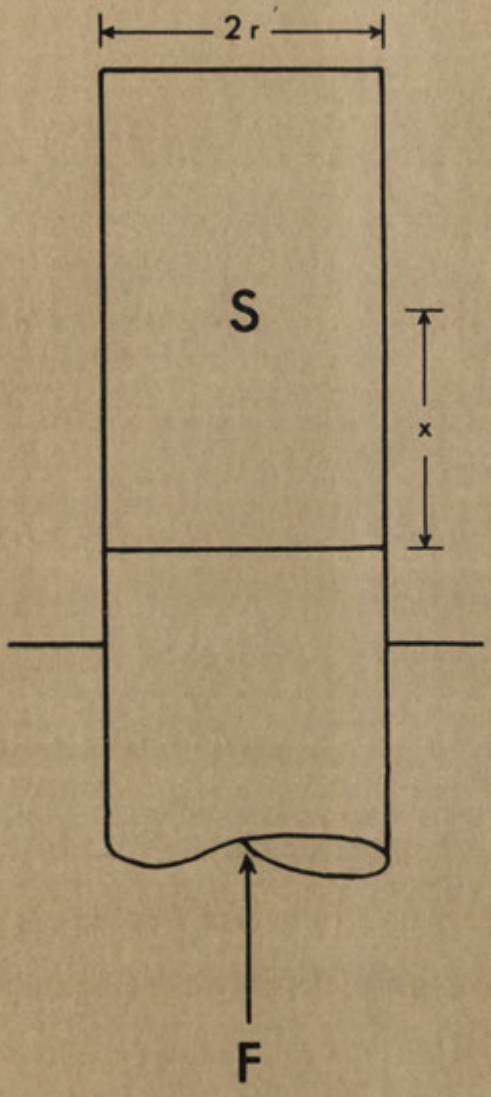


Fig. IX-2

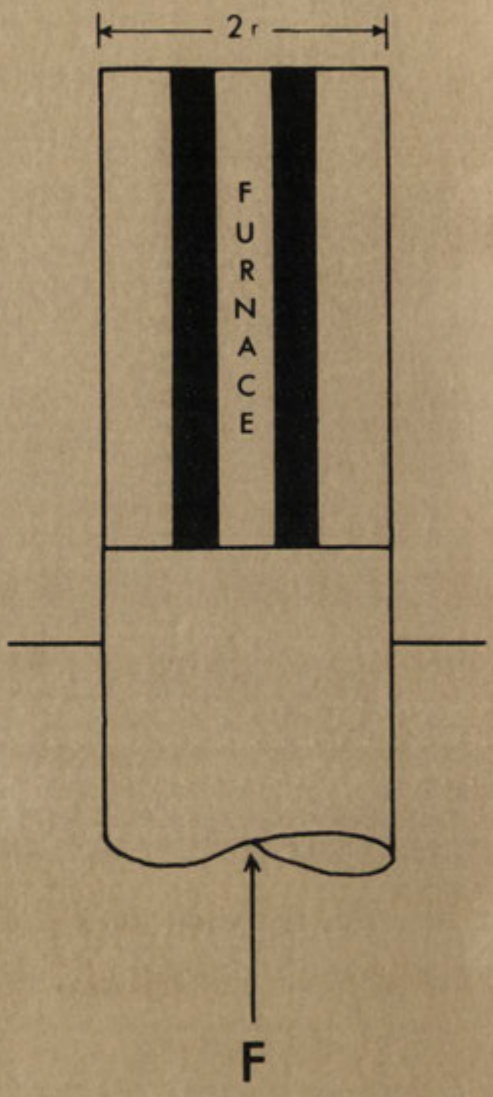


Fig. IX-3

CHAPTER X

GENERAL DISCUSSION

Increasing the confining pressure at which the materials tested in this thesis were deformed had six inter-related macroscopic effects:-

1. The levels of the stress-strain curves were generally raised.

2. Most of the materials underwent a brittle-ductile transition.

3. The rate of strain hardening increased.

4. The pressure sensitivity of the flow stress generally decreased.

5. Volume increases during deformation were progressively suppressed.

6. Volume increases during reduction of the confining pressure after deformation increased.

The brittle-ductile transition as we defined it in Chapter I is clearly a macroscopic phenomenon and does not by itself provide any specific information about what is happening at the microscopic level. Macroscopically it seems to occur when the stress required to cause sliding on a shear fracture becomes greater than the stress required to initiate the fracture, the former stress being more pressure sensitive than the latter. As pointed out in Chapter I, however, this is not a satisfactory explanation of the brittle-ductile transition, for while it may explain, albeit

criterion i.e.

$$\tau = \tau_0 + \sigma \tan \phi$$

where τ is the maximum shear strength

τ_0 is a constant (the cohesion of the material)

σ is the normal stress across the failure plane

$\tan \phi$ is the coefficient of internal friction on this plane.

Handin (1969) regarded this coefficient of internal friction as a "fictitious quantity" on the basis of the argument that until the cohesion is broken no sliding surface exists in the intact body. Immediately prior to failure, however, limiting friction must be reached on the surfaces of the cracks which make up the failure surfaces immediately after failure. Whilst it is conceded that the coefficient of internal friction in the Coulomb equation, and more generally the slope of the Mohr envelope, do include other than simple frictional components (such as components resulting from volume changes) it seems reasonable to assume that the coefficient of internal friction is closely related to the coefficient of friction on the failure surfaces immediately after failure.

It is suggested that the two most relevant differential stresses when considering cataclastic failure may be those required to (a) propagate cracks within the specimen σ_C

and (b) to overcome the frictional forces on those crack surfaces which are most favourably oriented σ_F. Both these stresses are sensitive to changes in confining pressure but it is generally observed that at high confining pressures the coefficient of friction tends to decrease. In a closed crack situation both σ_C and σ_F are functions of the coefficient of friction and their gradients may therefore be expected to decrease if they are plotted against the confining pressure (Fig. X-1). Unfortunately there is no clear information available to show exactly how σ_C will vary with confining pressure. Fig. X-1 may represent the failure conditions for a material having a brittle-ductile transition which is represented by the intersection X. In practice these boundaries would not be clear cut, but they would represent the middles of broad transition zones resulting from the heterogeneity of the material on the scale of the crystals. The relative positions of these boundaries would also vary from material to material. Paths such as A passing to the left of X will first exceed the stress required for sliding on crack surfaces. Most rock materials already contain cracks, particularly at grain boundaries, but in general there will not be a suitable macroscopic surface within the material composed entirely of crack surfaces on which sliding can occur. As the

differential stresses increase further, cracks in the material propagate, initially only in the most favoured orientations, along grain boundaries. With further increases in the stress, cracks will propagate in less favoured orientations until eventually one of an almost infinite number of suitable shear surfaces is composed of a continuous series of crack surfaces and macroscopic shear failure on this plane occurs with a stress drop which represents the difference between the stress required to propagate sufficient cracks and the stress required for sliding on the macroscopic surface. This stress difference as deduced from Fig. X-1 would not be a constant, even though it is directly related to the "cohesion" term in the Coulomb equation. Workers such as Byerlee (1967) have demonstrated that this "cohesion" term may not be a constant and that therefore the Coulomb equation is not generally applicable. Paths such as B passing to the right of X first exceed the stress required to propagate cracks and when the stress attains that required for macroscopic sliding, failure occurs. In this case no stress drop would occur, but the deformation would either be localised or distributed on the scale of the specimen depending on the influence of any strain hardening (or softening) mechanisms. The lack of a suitable strain hardening mechanism could

give rise to the so-called "ductile faulting" phenomenon when a fault is produced without a sudden stress drop (eg. Donath and Faill, 1963). Under conditions of cataclastic deformation, a stabilising effect may arise from comminution and changes of packing and also (as shown in Chapter III and using the same sign convention) from a negative value of the second derivative of the volume change with respect to the axial strain.

It is generally accepted that if the confining pressure is increased sufficiently, many materials which would otherwise deform cataclastically will undergo a transition to deformation by intra-crystalline plasticity. This transition is represented by the broken line in Fig. X-1 and occurs when the differential stress σ_I is attained, it being assumed that intra-crystalline mechanisms are insensitive to variations of the confining pressure. It is suggested that in cases where the transition to intra-crystalline plasticity requires a very high differential stress a ductile-brittle transition of the type described by Byerlee and Brace (1969) may occur at the intersection X' in Fig. X-1. Paths such as C would then be mechanistically similar to path A.

The volume change experiments reported in this thesis, if taken together with various earlier measurements

of volume changes by other workers, have shown that volume changes which are large compared with elastic effects can, and usually do, occur at some stage during the deformation of rocks and during the processes of increasing and decreasing the confining pressure. Let us therefore consider some of the implications of such volume changes.

It was noted in Chapter III that volume changes during deformation introduce a dependence on the intermediate principal stress. Most theoretical predictions of strength and failure completely ignore this and other effects of volume changes. Griffith's theory in both its original and modified (to include friction on the crack surfaces) forms is one of these. In its modified form, however, if the coefficient of friction is chosen empirically to fit the results then it will itself include a component which allows for volume change effects. In the case of volume increases the intrinsic coefficient of friction will be increased by an amount which is proportional to the product of the volume increase and the confining pressure. This may be the reason for the high friction coefficients often deduced for sliding on Griffith cracks.

In soil mechanics volume changes have long been considered of fundamental importance and recently the so-called "stress dilatancy theory" has been evolved (Rowe, 1962; Rowe, Barden and Lee, 1964; Lee and Ingles, 1968). The

theory is in fact of a semi-empirical nature. It appears to have some application in distinguishing between the different components of strength which make up the gross strength of a soil. The rigid body model and the simple friction mechanism postulated in this theory are very restrictive, however, and consequently the theory cannot be considered to have any application in cases where more mechanistic variety is found and where energy components which have been assumed negligible or ignored altogether are of importance. Application of this theory to results of my experiments led to nonsensical coefficients of friction even in the case of the Gosford sandstone where this theory may have been expected to have had most application.

A number of experimental studies have indicated that the presence of a pore pressure in rocks, as in soils, has a profound effect on strength. Hubbert and Willis (1957), Hubbert and Rubey (1959) and Jaeger (1962) predicted on theoretical grounds that the principal effective stresses should control rock strength, and this prediction has been verified by the experiments of Handin, Hager, Friedman and Feather (1963), Jaeger (1963) and Brace (1968). The so called "effective stress law" may be written:-

$$\bar{\sigma} = \sigma - u$$

where $\bar{\sigma}$ is the effective stress

σ is the total stress

u is the neutral stress or pore pressure.

Coulomb's equation may then be written

$$\tau = \tau_0 + \bar{\sigma} \tan \phi$$

Any non-elastic volume change of a rock causes a change in the porosity. The pressure of a pore fluid such as water which is relatively incompressible is very sensitive to such changes of porosity. For example the pressure of water is increased from zero to about 10 kb by a 20% decrease in its volume. Volume decreases may therefore cause sharp increases in the pore pressure and consequent reductions in the effective stresses and the strength of the rock.

Conversely, volume increases can cause a sharp reduction in the pore pressure and increases in the strength of the rock. This effect is commonly known as "dilatancy hardening" and was demonstrated by Reynolds (1885). Mead (1925) discussed the geological role of dilatancy, and Frank (1965) suggested that dilatancy could give rise to a mechanical instability of the type needed to account for seismic faulting. It is interesting to note that Frank's criterion for instability which according to my sign convention requires that $d^2v/d\nu^2 > 0$, where v is the volume and ν is the shear strain, is closely related to my equation -(2) on page III-13 which leads to a strain softening effect if

$d^2v/d\varepsilon_A^2 > 0$ even in the absence of a pore fluid. Orowan (1966), in reply to Frank (1965), pointed out that this type of instability is also a familiar property of compacted granular masses quite independent of any volume changes. Orowan's criticism of Frank's paper on the basis of this very restrictive model was not justified, however, because under the conditions Frank was considering this type of instability does not automatically occur during deformation as Orowan suggested. My own results demonstrate that $d^2v/d\varepsilon_A^2$ can take on values of both signs.

The results of laboratory triaxial experiments performed at room temperature and at relatively fast strain rates in geological terms are not likely to be directly applicable to geological situations where the state of stress may vary in a more complex manner, and high temperatures and low strain rates may prevail. Volume changes during deformation provide a very versatile mechanism for the manipulation of pore pressures as required for example by Hubbert and Rubey (1959) to cause low angle overthrust faulting, and by Secor (1965, 1968) to cause extension fracturing at depth in the earth's crust. They also provide a mechanism by which fluids may be sucked into zones of deformation. Such processes would be further assisted by increases in the permeability which may result from increases in porosity

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either during deformation or during later reductions of the hydrostatic pressure component.

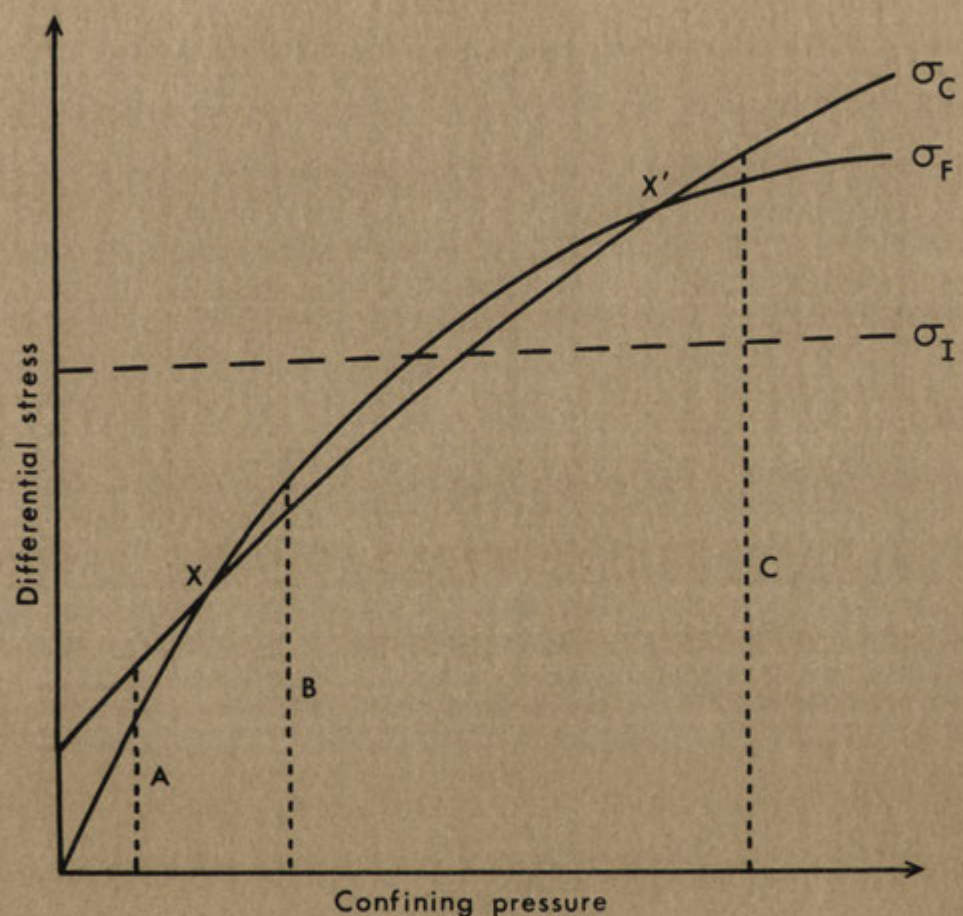

Fig. X-1 Suggested failure conditions for a material having a brittle-ductile transition.

APPENDIX

1. Load bearing capacity of 10 mm inside diameter, 1.6mm wall thickness red rubber jackets at room temperature

No correction was applied for the load bearing capacity during experiments at confining pressures of 4 kb or less. These pressures were well below the room temperature glass transition pressure of the rubber (Paterson, 1964b; Weaver and Paterson, 1969). Other experiments performed using these jackets were all at a confining pressure of 8 kb. Two annealed copper specimens (10 mm diameter and 20 mm long) were deformed at 8 kb without jackets and two were deformed at the same confining pressure with rubber jackets. Both pairs of experiments showed good reproducibility and the difference in load bearing capacity between the means of each pair was taken as the load bearing capacity of the rubber jacket.

Strain (%)	Load borne by jacket (kg)
1	280
2	350
5	380
10	380
15	410
20	490

2. Load bearing capacity of 10 mm inside diameter, 0.25 mm wall thickness annealed copper jackets at room temperature

These figures were determined using the same method used for the rubber jackets. The same corrections were used at all confining pressures.

Strain (%)	Load borne by jacket (kg)
1	30
2	50
5	100
10	140
15	150
20	155

It was found that this method gave marginally lower corrections at strains greater than 10% than the method of multiplying the load bearing capacity of the unjacketed annealed copper specimen by the ratio of the cross sectional area of the jacket to that of the specimen.

3. Load bearing capacity of 7 mm inside diameter, 0.25 mm wall thickness annealed copper jackets at room temperature

These figures were obtained by deforming in extension three jacketed annealed copper specimens (7 mm diameter and 13 mm long) and multiplying the load bearing capacity of the jacketed specimen by the ratio of the cross sectional area of the jacket to that of the specimen plus the jacket.

Strain (%)	Load borne by jacket (kg)
1	25
2	50
5	80
10	120
15	130
20	140

4. Load bearing capacity of 10 mm inside diameter, 0.25 mm wall thickness annealed copper jackets at high temperatures

Experiments by Paterson (unpublished data) in which specimens of annealed copper were deformed at high temperatures showed that the load bearing capacity of these copper jackets could be obtained very closely by multiplying the load bearing capacity of the copper jackets at room temperature in section 2 above by a "temperature factor" which is independent of strain.

Temperature (^{o}C)	Temperature factor
25	1.00
200	0.77
300	0.65
400	0.52
500	0.40
600	0.28

No correction was applied for the load bearing capacity of the copper jackets at temperatures of 700^{o}C and above.

5. Effect of jackets on specimen behaviour

Several experiments were performed with talc and graphite using rubber jackets, the results of which have not been included in this thesis because they were repeated later using copper jackets during volume change experiments. It was a general observation with these materials however, that the brittle-ductile transition occurred at a slightly lower confining pressure when copper jackets were used. This effect may be due to the greater strength of the copper jackets under those conditions inhibiting localised deformation.

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